

PB-223 051

GUIDELINES FOR DEVELOPMENT OF A QUALITY ASSURANCE
PROGRAM

RESEARCH TRIANGLE INSTITUTE

PREPARED FOR
ENVIRONMENTAL PROTECTION AGENCY

JUNE 1973

DISTRIBUTED BY:

NTIS

National Technical Information Service
U. S. DEPARTMENT OF COMMERCE

EPA-R4-73-028b

PB 223 051

June 1973

Environmental Monitoring Series



Reproduced by
**NATIONAL TECHNICAL
INFORMATION SERVICE**
U.S. Department of Commerce
Springfield VA 22151

BIBLIOGRAPHIC DATA SHEET		1. Report No. EPA-R4-73-028b	2.	3. Recipient's Accession No. PB-823 651
4. Title and Subtitle GUIDELINES FOR DEVELOPMENT OF A QUALITY ASSURANCE PROGRAM Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method)				5. Report Date June 1973
7. Author(s) Franklin Smith and A Carl Nelson, Jr.				8. Performing Organization Rept. No.
9. Performing Organization Name and Address Research Triangle Institute Research Triangle Park, North Carolina 27709				10. Project/Task/Work Unit No.
12. Sponsoring Organization Name and Address Environmental Protection Agency National Environmental Research Center Research Triangle Park, North Carolina 27711				11. Contract/Grant No. EPA- Durham 68-02-0598
				13. Type of Report & Period Covered Interim Contract Report - Field document
15. Supplementary Notes				14. /
16. Abstracts Guidelines for quality control of ambient suspended particulate measurements by the Federal reference method are presented. These include: 1. Good operating practices 2. Directions on how to assess data and qualify data 3. Directions on how to identify trouble and improve data quality 4. Directions to permit design of auditing activities 5. Procedures which can be used to select action options and relate them to costs This document is not a research report. It is designed for use by operating personnel.				
17. Key Words and Document Analysis. 17a. Descriptors Quality Assurance Quality Control Air Pollution Quantitative Analysis Aerosols 17b. Identifiers/Open-Ended Terms 17c. COSATI Field/Group 13H, 14D, 07D, 13B				
18. Availability Statement		19. Security Class (This Report) UNCLASSIFIED		21. No. of Pages 130
		20. Security Class (This Page) UNCLASSIFIED		22. Price 4.50

GUIDELINES FOR DEVELOPMENT OF A QUALITY ASSURANCE PROGRAM

Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method)

by

Franklin Smith and A. Carl Nelson, Jr.

Research Triangle Institute
Research Triangle Park, North Carolina 27709

Contract No. 68-02-0598
Program Element No. 1H1327

EPA Project Officer: Dr. Joseph F. Walling

Quality Assurance and Environmental Monitoring Laboratory
National Environmental Research Center
Research Triangle Park, North Carolina 27711

Prepared for

OFFICE OF RESEARCH AND MONITORING
U.S. ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

June 1973

This report has been reviewed by the Environmental Protection Agency and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

PREFACE

Quality control is an integral part of any viable environmental monitoring activity. The primary goals of EPA's quality control program are to improve and document the credibility of environmental measurements. To achieve these goals, quality control is needed in nearly all segments of monitoring activities and should cover personnel, methods selection, equipment, and data handling procedures. The quality control program will consist of four major activities:

- Development and issuance of procedures
- Intra-laboratory quality control
- Inter-laboratory quality control
- Monitoring program evaluation and certification

All these activities are essential to a successful quality control program and will be planned and carried out simultaneously.

Accordingly, this second manual of a series of five has been prepared for the quality control of ambient air measurements. These guidelines for the quality control

of suspended particulate measurements in the atmosphere have been produced under the direction of the Quality Control Branch of the Quality Assurance and Environmental Monitoring Laboratory of NERC-RTP. The purpose of this document is to provide uniform guidance to all EPA monitoring activities in the collection, analysis, interpretation, presentation, and validation of quantitative data. In accordance with administrative directives to implement an Agency-wide quality control program, all EPA monitoring activities are requested to use these guidelines to establish intra-laboratory quality assurance programs in the conduct of all ambient air measurements for suspended particulates. Your comments on the utility of these guidelines, along with documented requests for revision(s), are welcomed.

All questions concerning the use of this manual and other matters related to quality control of air pollution measurements should be directed to:

Mr. Seymour Hochheiser, Chief
Quality Control Branch
Quality Assurance and Environmental
Monitoring Laboratory
National Environmental Research Center
Research Triangle Park, North Carolina 27711

Information on the quality control of other environmental media and categorical measurements can be obtained by contacting the following person(s):

Water

Mr. Dwight Ballinger, Director
Analytical Quality Control Laboratory
National Environmental Research Center
Cincinnati, Ohio 45268

Pesticides

Dr. Henry Enos, Chief
Chemistry Branch
Primate and Pesticide Effects Laboratory
Environmental Protection Agency
Perrine, Florida 33157

Radiation

Mr. Arthur Jarvis, Chief
Office of Quality Assurance-Radiation
National Environmental Research Center
Las Vegas, Nevada 89114

During the months ahead, a series of manuals will be issued which describe guidelines to be followed during the course of sampling, analysis, and data handling. The use of these prescribed guidelines will provide a uniform approach in the various monitoring programs which allows the evaluation of the validity of data produced. The implementation of a total and meaningful quality control program cannot succeed without the full support of all monitoring programs. Your cooperation is appreciated.

TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
1.0 INTRODUCTION	1
PART I. OPERATIONS MANUAL	3
2.0 GENERAL	3
2.1 Operating Procedures	6
FILTER SELECTION AND PREPARATION	6
SAMPLE COLLECTION	8
SAMPLE ANALYSIS	22
DATA PROCESSING	23
2.2 Flow Rate Calibration	24
2.3 Relative Humidity Indicator Calibration	34
2.4 Analytical Balance Calibration	34
2.5 Elapsed Time Indicator Check	34
2.6 Special Checks for Auditing Purposes	35
2.7 Special Checks to Detect and Identify Trouble	40
2.8 Maintenance	43
2.9 Facility and Apparatus Requirements	44
PART II. SUPERVISION MANUAL	49
3.0 GENERAL	49
3.1 Assessment of High Volume Data	51
3.1.1 Assessment by Auditing Individual Variables	51
3.1.2 Assessment by Auditing With a Mobile Sampler	60

TABLE OF CONTENTS (Concl'd)

<u>Section</u>	<u>Page</u>
3.2 Suggested Standards for Judging Performance Using Audit Data	61
3.2.1 Suggested Performance Standards for Variables	61
3.2.2 Suggested Standards for Comparing With Mobile Sampler	61
3.3 Collection of Information to Detect and Identify Trouble	63
3.3.1 Identification of Important Variables	63
3.3.2 How to Monitor Important Variables	72
3.3.3 Suggested Control Limits	72
3.4 Procedures for Improving Data Quality	74
3.5 Procedures for Changing the Auditing Level to Give the Desired Level of Confidence in the Reported Data	77
3.6 Monitoring Strategies and Cost	78
PART III. MANAGEMENT MANUAL	81
4.0 GENERAL	81
4.1 Data Quality Assessment	82
4.2 Auditing Schemes	89
4.3 Data Quality Versus Cost of Implementing Actions	103
4.4 Data Presentation	109
4.5 Personnel Requirements	111
4.6 Operator Proficiency Evaluation Procedures	112
REFERENCES	115
APPENDIX A: REFERENCE METHOD FOR THE DETERMINATION OF SUSPENDED PARTICULATES IN THE ATMOSPHERE (HIGH VOLUME METHOD)	A-1

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Sequence of Operations Required in the High Volume Method	4-5
2	Servicing Position of High Volume Sampler	9
3	Operating Position of High Volume Sampler	9
4	Examples of Nonuniform Border Resulting from Poorly Aligned Filters	10
5	Flow-rate Recorder with Chart Installed	13
6	Example of Smudged Borders Resulting from Improperly Folded Filters	17
7	Examples of Air Leaks Around the Filter Due to a Worn Faceplate Gasket	18
8	Properly Prepared Filter Folder and Accompanying Filter Mat with Recorder Chart	21
9	Typical Field Calibration Setup for Modified High Volume Sampler	27
10	Percent Change in Flow Rate Versus Temperature Variations (Orifice Calibration Unit)	29
11	Sample Calibration Sheet	31
12	Flow Chart of Quality Control Checks in the Auditing Program	52
13	Data Qualification Form	59
14	Particulate Concentration and Flow Rate as Functions of Time	71
15	Symmetrical Diurnal Concentration Pattern	71
16	Critical Values of Ratio s_i/σ_i Vs n	86
17	Data Flow Diagram for Auditing Scheme	92
18A	Probability of d Defectives in the Sample if the Lot (N = 100) Contains D% Defectives	93
18B	Probability of d Defectives in the Sample if the Lot (N = 50) Contains d% Defectives	94

LIST OF FIGURES (Concl'd)

<u>Figure</u>		<u>Page</u>
19A	Percentage of Good Measurements Vs. Sample Size for No Defectives and Indicated Confidence Level	96
19B	Percentage of Good Measurements Vs. Sample Size for 1 Defective Observed and Indicated Confidence Level	97
20	Average Cost Vs. Audit Level	104
21	Added Costs Vs. $\sqrt{\sigma_T^2 + \tau^2}$ for Alternative Strategies	108
22	Sample QC Chart for Evaluating Operator Proficiency	113

LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	Sampling Site Evaluation Data	41
2	Apparatus Used in the High Volume Method	47
3	Suggested Performance Standards	62
4	Methods of Monitoring Variables	73
5	Suggested Control Limits for Parameters and Variables	74
6	Quality Control Procedures or Actions	75
7	Critical Values of s_1/σ_1	85
8	P(d defectives)	91
9	Required Auditing Levels n for Lot Size N = 100 Assuming Zero Defectives	95
10	Costs vs. Data Quality	98
11A	Costs If 0 Defectives are Observed and the Lot is Rejected	99
11B	Costs If 0 Defectives are Observed and the Lot is Accepted	99
12	Costs in Dollars	100
13	Overall Average Costs for One Acceptance - Rejection Scheme	102
14	Assumed Standard Deviations and Biases for Alternative Strategies	107

ABSTRACT

Guidelines for the quality control of total suspended particulate measurements by the Federal reference method are presented. These include:

1. Good operating practices
2. Directions on how to assess data and qualify data
3. Directions on how to identify trouble and improve data quality
4. Directions to permit design of auditing activities
5. Procedures which can be used to select action options and relate them to costs

The document is not a research report. It is designed for use by operating personnel.

This work was submitted in partial fulfillment of Contract Durham 68-02-0598 by Research Triangle Institute under the sponsorship of the Environmental Protection Agency. Work was completed as of May 1973.

1.0 INTRODUCTION

This document presents guidelines for implementing a quality assurance program for measuring the mass concentration of suspended particulates using the High Volume Method.

The objectives of this quality assurance program for the High Volume Method of measuring suspended particulates are to:

- 1) provide routine indication, for operating purposes, of unsatisfactory performance of personnel and/or equipment.
- 2) provide for prompt detection and correction of conditions which contribute to the collection of poor quality data, and
- 3) collect and supply information necessary to describe the quality of the data.

To accomplish the above objectives, a quality assurance program must contain the following components:

- 1) routine training and evaluation of operators,
- 2) routine monitoring of the variables and parameters which may have a significant effect on data quality,
- 3) development of statements and evidence to qualify data and detect defects, and
- 4) action strategies to increase the level of precision in the reported data and/or to detect instrument defects or degradation and to correct same.

Implementation of a quality assurance program will result in data that are more uniform in terms of precision and accuracy. It will enable each monitoring network to continuously generate data that approach the highest level of accuracy attainable with the High Volume Method.

This document is divided into three parts. They are:

Part I, Operations Manual - The Operations Manual sets forth recommended operating procedures, instructions for performing control checks designed to give an indication or warning that invalid or poor quality data are being collected, and instructions for performing certain special checks for auditing purposes.

Part II, Supervision Manual - The Supervision Manual contains directions for 1) the assessment of high volume data, 2) collection of information to detect and/or identify trouble, 3) applying quality control procedures to improve data quality, and 4) varying the auditing or checking level to achieve a desired level of confidence in the validity of the outgoing data. Also, monitoring strategies and costs as discussed in Part III are summarized in this manual.

Part III, Management Manual - The Management Manual presents procedures designed to assist the manager in 1) detecting when data quality is inadequate, 2) assessing overall data quality, 3) determining the extent of independent auditing to be performed, 4) relating costs of data quality assurance procedures to a measure of data quality, and 5) selecting from the options available the alternative(s) which will enable him to meet the data quality goals by the most cost-effective means. Also, discussions on data presentation and personnel requirements are included in this manual.

The scope of this document has been purposely limited to that of a field document. Additional background information is contained in the final report under this contract.

PART I. OPERATIONS MANUAL

2.0 GENERAL

This Operations Manual sets forth recommended operating procedures for measuring the mass concentration of suspended particulates using the High Volume Method. Quality control procedures and checks designed to give an indication or warning that invalid or poor quality data are being collected are written as part of the operating procedures, and are to be performed by the operator on a routine basis. In addition, the performance of special quality control procedures and checks as prescribed by the supervisor may be required of the operator on certain occasions.

The sequence of operations to be performed is given in Figure 1. Two columns are used. The first column numbering 1 through 16 gives the operating procedures in sequential order as one filter progresses through the system. Calibration procedures that are performed periodically are given in the second column. In general, Steps 1 through 7 and 12 through 16 are carried out in the laboratory, and Steps 8 through 11 are performed at the sampling site. Quality checkpoints in the measurement process for which appropriate quality control limits are assigned are represented by blocks enclosed by heavy lines. Other checkpoints involve go/no-go checks and subjective judgments by the operator with proper guidelines for decision making spelled out in the procedures. Under normal conditions, all calibrations are performed in the laboratory. (Additional calibrations in the field, however, may be advantageous in certain situations.) Instructions for performing each operation are presented in the same order as they appear in Figure 1. Calibration procedures follow the operating procedures and are numbered as in Figure 1.

The accuracy and/or validity of data obtained from this method depends upon instrument performance and the proficiency with which the operator performs his various tasks. Deviations from the recommended operational procedure may result in the collection of invalid data or at least reduce the quality of the data. The operator should become familiar with the

FILTER SELECTION AND PREPARATION

1. Select filters meeting specification of reference method. Analyze for surface alkalinity.

2. Visual inspection of filters for pinholes and other imperfections.

3. Permanently mark each filter with a serial number.

4. Equilibrate filter in conditioning environment 24 hours.

5. Check balance and weigh filter to the nearest mg.

6. Record filter serial number and tare weight in laboratory log book.

7. Package filter for shipping or storage.

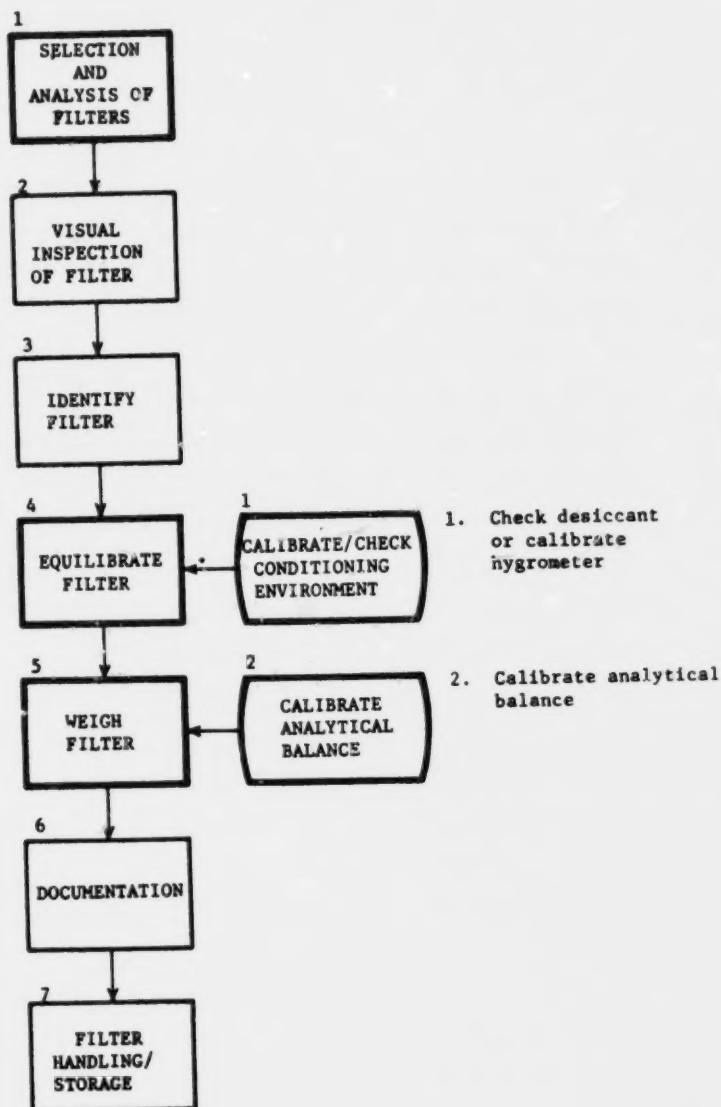


Figure 1: Sequence of Operations Required in the High Volume Method

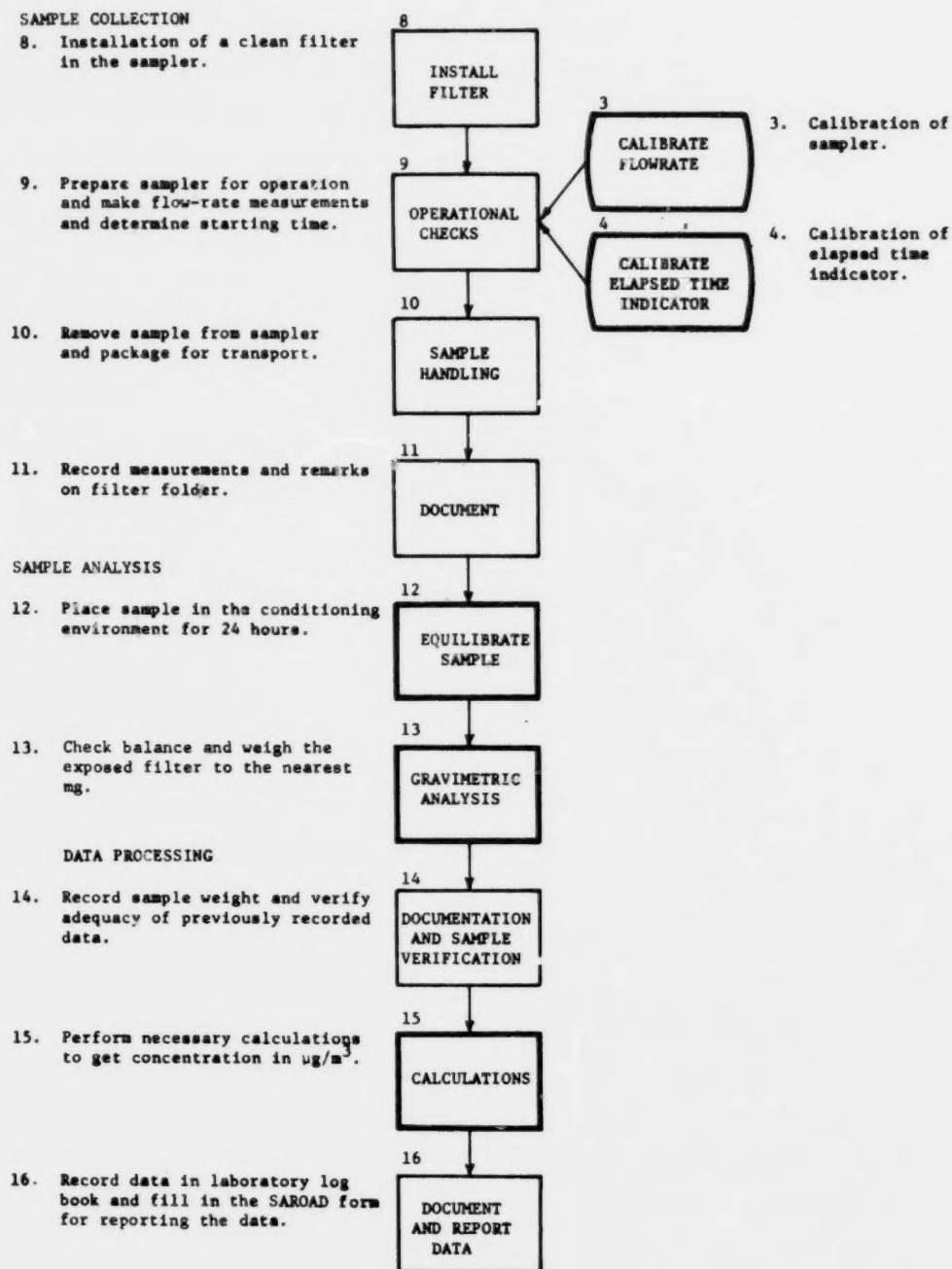


Figure 1: Sequence of Operations Required in the High Volume Method (Cont'd)

manufacturer's operational instructions and with the rules and regulations concerning the High Volume Method as written in the Federal Register, Vol. 36, No. 84, Part II, April 30, 1971 (see Appendix A of this document).

The operator is responsible for maintaining certain records. Specifically log books must be maintained in the laboratory for recording (1) filter processing data (i.e., tare weight, serial number, sampling station, etc.), (2) calibration data (past calibration data and future calibration schedules), and (3) maintenance information including a historical record and future schedule. A site log book is maintained by the operator and kept in the sampler shelter. This log book has the most recent calibration data and schedules for future maintenance and calibrations. Initial and final flow rates are recorded in the log book for each sampling period.

All directions are written for a nonautomated system. If an automatic data management system is used, certain of these operations will be performed automatically.

2.1 Operating Procedures

FILTER SELECTION AND PREPARATION

Step 1. Selection of Filter Media

A. Filter Collection Efficiency

Only filters having a collection efficiency of at least 99 percent, for particles of 0.3 μm diameter, as measured by the DOP test, should be used. The manufacturer should be required to furnish proof of the collection efficiency of a batch of new filters when purchased.

B. Filter Surface Alkalinity

It is recommended that only filters with a surface alkalinity between 6.5 and 7.5 on the pH scale be used. Surface alkalinity for a new batch of filters can be determined by performing the analysis as described in A of Section 2.6.

Step 2. Visual Inspection of Filter

Each filter must be visually inspected with the aid of a light table. Look for pinholes, loose particles and other defects such as tears, creases or lumps. Remove loose particles with a soft brush. Filters with other imperfections should be destroyed.

Step 3. Filter Identification

Assign a serial number to each filter. Stamp this number on two diagonally opposite corners on opposite sides of the filter using a numbering device. Apply gentle pressure to avoid damaging the filter.

Step 4. Filter Equilibration

Equilibrate the filter in the conditioning environment (see Section 2.9 for a description of conditioning chamber and environment) for 24 hours prior to weighing. This is necessary to avoid a significant error in measuring the weight of the filter.

Step 5. Filter Weighing

Clean filters are usually processed in lots, that is, several at one time. Before weighing the first filter, perform a balance check by weighing a standard weight of between 3 and 5 grams.

Record the actual and measured weights in the laboratory log book along with the date and operator's initials.

If the actual and measured values differ by more than $\pm .5$ mg (0.0005g), report it to the supervisor before proceeding.

If the actual and measured values agree to within $\pm .5$ mg, proceed to weigh each filter to the nearest mg. Clean filters must not be folded for weighing. A special balance pan is required to accommodate 20.3 by 25.4 cm filters.

Step 6. Documentation

Record the tare weight and serial number of each filter in the laboratory log book.

Step 7. Filter Handling

Place the weighed filters in a folder or suitable container to protect them from damage before use. Filters must not be folded or creased prior to use.

Supply weighed filters, filter folders, glassine envelopes and suitable mailing envelopes to each sampling station operator as required.

SAMPLE COLLECTION

Step 8. Installation of Clean Filter

To facilitate filter installation, place the sampler in the servicing position as illustrated in Figure 2 (Figure 3 shows the normal operating position). Place the sampler in the servicing position by raising the sampler until the filter holder is above the top level of the shelter; then rotate the unit one-eighth turn so that the motor assembly hangs from the top of the filter holder. During inclement weather (i.e., rain, snow, sleet, or high winds), it is suggested that the sampler be removed completely to a protected area. Extreme care should be exercised to prevent damage to the clean filter during this operation.

Remove the faceplate by loosening the four wing nuts and rotating the bolts outward. Place the filter, rough side up, on the wire screen. Center the filter on the screen so that when the faceplate is in position, the gasket will form an airtight seal on the outer edge (1/2 inch) of the filter. When aligned correctly, the edges of the filter should be parallel both to the edges of the screen behind it and to the faceplate gasket above it. The results of poorly aligned filters are shown in Figure 4. Note the uneven white border around the filter. Results of a correctly aligned filter can be seen in Figure 8 on page 21.

Once the filter is aligned and the faceplate is in place, the four wing nuts are tightened so that the gasket is airtight against the filter. Tighten diagonally opposite wing nuts first to prevent distortion of the cast iron frame, and to give a more even tightening of the wing nuts.

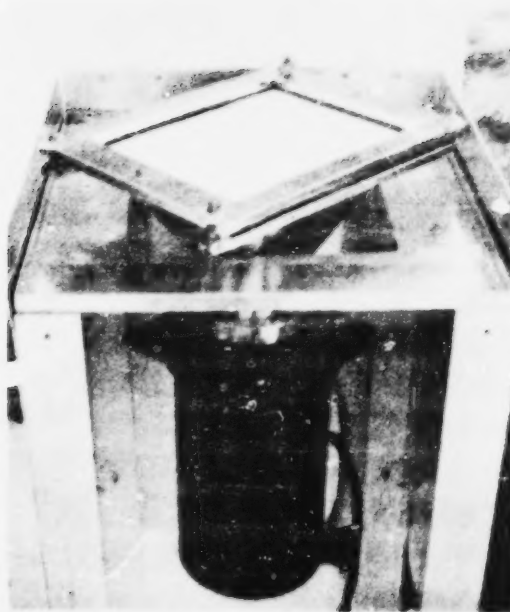


Figure 2: Servicing Position of High Volume Sampler

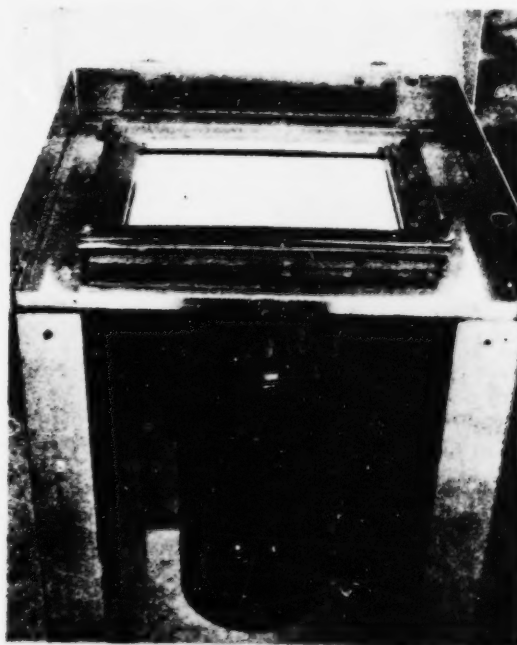


Figure 3: Operating Position of High Volume Sampler

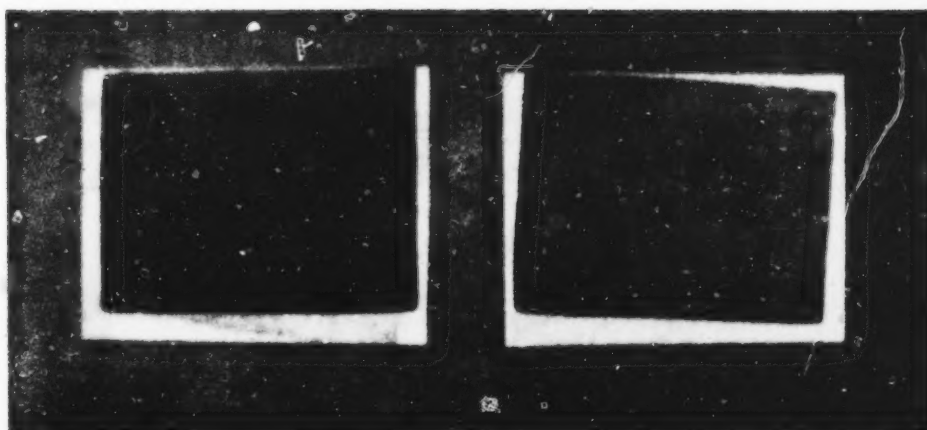


Figure 4 : Examples of Nonuniform Border Resulting from
Poorly Aligned Filters

Excessive tightening of the wing nuts should be avoided to help minimize the tendency of the filter to stick to the gasket and to guard against permanent damage to the gasket itself.

The entire motor assembly (sampler) is rotated and lowered to its normal operating position as shown in Figure 3.

Also, while the sampler is removed from the shelter or before the new filter is installed, the inside surfaces of the shelter lid and area around the filter holder should be cleaned of loose particles by wiping with a clean rag.

Step 9. Operational Checks

A. Flow-Rate Measurements

1. Sampler Equipped With Rotameter

Make flow-rate measurements while the sampler is at normal operating temperature. This requires a warmup time of at least 5 minutes before a valid measurement can be obtained.

Connect the rotameter to the sampler, using the same tubing as was used to calibrate, and place or hold in a vertical position at eye level. Read the widest part of the float. Use the calibration chart to convert the reading to cubic meters per minute rounded to the nearest $0.03 \text{ m}^3/\text{min}$ (see Section 2.2 for use of calibration chart).

Flow rates are measured at the beginning and end of a sampling period. Additional measurements at different times during the sampling period may be required in special instances.

Precautions to be taken when making flow-rate measurements include:

- a) After connecting the rotameter to the sampler, observe for at least one minute before taking a reading. If a gradual change in flow rate is observed do not take a reading until an equilibrium is reached. A gradual change will usually be observed when the rotameter is at a substantially different temperature from the sampler exhaust air and may require 2 to 3 minutes to equilibrate.

- b) If a clock switch is used to start and stop the sampler at preset times, in order to minimize errors due to weather changes or changes in the collected particulates, it may be necessary to make flow-rate measurements within 30 minutes of the actual start and stop times.
2. Sampler Equipped With Continuous Flow Recorders
- Prepare the recorder for operation as follows:
- a) Record on the backside of the new chart the filter number, station and sampler numbers, start time, and date of start time.
 - b) Remove any moisture from inside the recorder case by wiping with clean cloth. Carefully insert the new chart into the recorder, being careful not to bend the pen arm beyond its limits of travel. An easy way to do this is to push in on the extreme top of the pen arm with the right hand to raise the pen head while inserting the chart with the left hand. A properly installed chart is shown in Figure 5. Be careful not to damage or weaken the center tab on the chart and make certain that the tab is centered on the slotted drive so that the chart will rotate the full 360 degrees in 24 hours with no binding or slippage.
 - c) Check to see that the pen head rests on zero (i.e., the smallest diameter circle on the chart). If it does not, tap the recorder lightly to make certain the pen arm is free; if it still does not read zero, adjust to zero with the adjustment screw (follow manufacturer's direction for specific recorder).

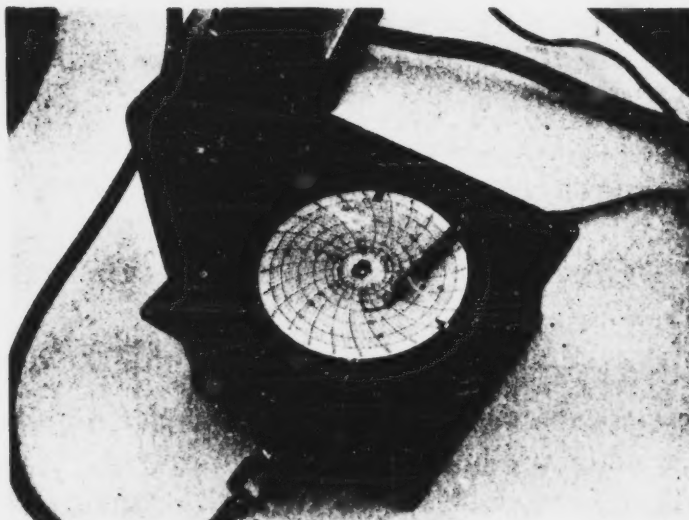


Figure 5: Flow-rate Recorder with Chart Installed

- d) Check the time indicated by the pen. If it is in error, rotate the chart in a clockwise direction, by inserting a screwdriver or coin into the slotted drive in the center of the chart face, until the correct time is indicated. Remember that if the sampler is started with a clock switch, the correct time for the recorder chart is the starting time on the clock switch.
- e) With an eyedropper put a small amount of ink into the hole in back of the pen tip.
- f) Turn on sampler (never turn on the sampler unless a filter is in place or the transducer and recorder may be damaged) and observe long enough to determine whether the transducer and recorder are operating properly.
- g) Turn off sampler and set the clock switch for correct start and stop times.

3. Routine Flow-Rate Checks

Record the initial and final flow-rate readings for each sample in the log book maintained in the sampler.

After each calibration, average the first four initial flow-rate measurements. Future initial flow rates deviating more than ± 10 percent from this average should be investigated. If the change has been gradual over a period of time a calibration is required.

When large deviations occur between successive samples, the operator should wait 5 minutes and make an additional reading. If the second reading falls within ± 10 percent of the average, continue normal operation. If the second reading falls out of bounds, 1) check the line voltage, and/or 2) replace the filter. A calibration check is made if neither of the above checks identifies the trouble (see Section 2.6 for instructions on performing calibration check). Continue normal operations if the calibration check is satisfactory, and perform a complete calibration if the check is unsatisfactory (see Section 2.2 for calibration procedures). Samplers equipped with a continuous recorder should be observed for at least 5 minutes before recording the initial flow rate.

The same procedure is used for final flow-rate measurements except that a larger range, say ± 20 percent of the average, should be used. Valid limits can be determined for each sampling site as data are available. A final flow rate deviating from the average by more than 20 percent may result from short-term inversions and humidity fluctuations. The occurrence of such conditions is noted on the data sheet for that sample. If a final flow rate less than $0.57 \text{ m}^3/\text{min}$ ($20 \text{ ft}^3/\text{min}$) is observed, the sample is voided because at this low air flow the motor heats up and a valid flow-rate measurement cannot be obtained.

B. Time Measurements

Sampling period start and stop times, for samplers not equipped with a clock switch or elapsed time meter, are determined by the operator who starts the sampler and the operator who stops the sampler respectively. If different operators are involved, they set their watches to a common reference in order to arrive at an accurate sampling period time. Such a reference could be an office clock which is checked daily or the local telephone company giving time-of-day service.

Start and stop times for samplers equipped with a clock switch are taken from the clock settings. The clock is checked, and set if necessary, for the correct time at each filter change. These clocks cannot be set or read to less than ± 15 minutes and, therefore, must be accompanied by an elapsed time indicator accurate to at least ± 4 minutes for a 24-hour period to satisfy the reference method specifications. The elapsed time is recorded on the filter folder along with the start and stop times.

For samplers equipped with neither an elapsed time indicator nor a continuous flow-rate recorder, the local power company should be contacted to see if the power has been off anytime during the sampling period. If so, the length of time that the power was off and the source of the information are recorded on the filter folder on the line for remarks.

Samples should be voided when the sampling period is less than 23 hours or greater than 25 hours.

Step 10. Sample Handling

A. Removing Exposed Filter

Place the sampler in the servicing position (see Figure 2). Remove the faceplate and remove the exposed filter from the supporting screen by grasping it gently at the ends (not at the corners) and lifting it from the screen. Fold the filter lengthwise at the middle, with the exposed side in. If the collected sample is not centered on the filter (i.e., the unexposed border is not uniform around the filter) fold the filter accordingly so that sample touches sample only. Results of an improperly folded filter are illustrated in Figure 6, the smudge marks can be seen extending across the right-hand border. This renders the sample useless for certain analyses where the collected sample has to be subdivided into equal portions.

Place the filter in a filter folder and glassine envelope and then in a mailing envelope if the sample is to be mailed to the laboratory.

For samplers equipped with a flow-rate recorder, the associated recorder chart is removed (see instructions for installing chart, Section 2b of Step 9), the stop time is recorded on the backside, and the chart is placed inside the filter folder with the inked side against the filter folder and the back (clear) side against the filter. This prevents ink from getting on the filter and interfering with future chemical analyses.

B. Routine Checks

The following checks should be made when removing an exposed filter.

- 1) Check the filter for signs of air leakage. Leakage may result from a) a worn faceplate gasket as illustrated in Figure 7, b) an improperly installed gasket as illustrated in Figure 4, or c) over-tightening of the faceplate gasket, cutting the filter along the gasket interface.

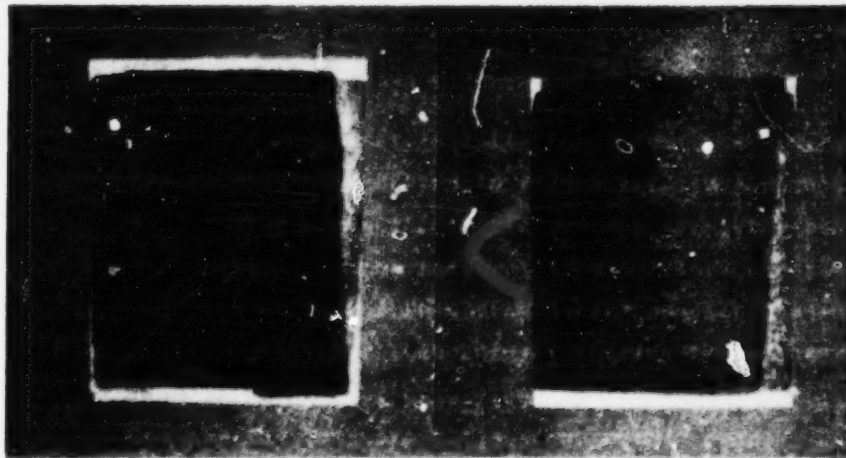


Figure 6: Example of Smudged Borders Resulting from Improperly Folded Filters, Leaking Gasket and Poor Alignment

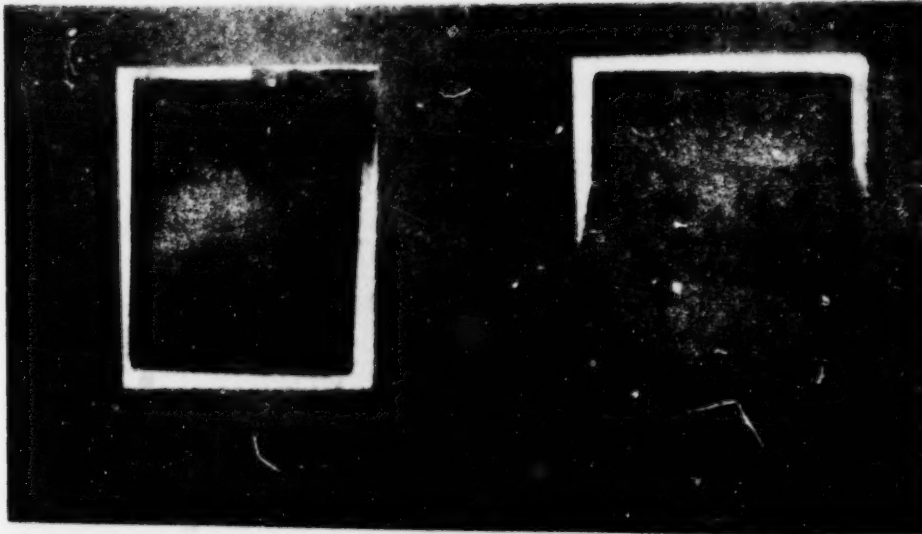


Figure 7: Examples of Air Leaks Around the Filter Due to a Worn Faceplate Gasket or Improper Installation

If at any time a leakage is observed, void the sample and take corrective action before starting another sampling period. Corrective action would be to replace the gasket, take more care in installing the filter, or applying more caution in tightening the gasket according to the cause of the leakage. Generally a gasket deteriorates slowly and the operator can tell well in advance, by an increasing fuzziness of the sample outline, to change the gasket before a total failure results.

- 2) Visually inspect the gasket face to see if glass fibers from the filter are being left behind. This is a sign of over-tightening the gasket. Tighten the gasket just enough to prevent leakage.
- 3) The operator should check the exposed filter for physical damage that may have occurred during sampling or after sampling. Physical damage to the filter after the sample has been collected does not always invalidate the sample. For example, accidentally tearing a corner off while removing the filter does not invalidate the sample if all pieces of the filter are included in the folder. However, any loss of sample due to leakages during the sampling period or to the loss of loose particulates from the filter after sampling (e.g., loss of particulates when folding the filter) invalidates the sample. The operator should mark all such samples void and forward them to the laboratory. Bugs such as gnats loosely attached to the filter should be removed by hand or with teflon-tipped tweezers. If they are embedded in the particulates note this on the folder and do not try to remove them.

- 4) The appearance of the particulates should be checked. Any changes from the normal color, for example, may indicate new emission sources or construction activity in the area, etc. The change should be noted on the filter folder along with any obvious reasons, if there are any, for the change.

Step 11. Documentation

In most instances the filter folder is the only immediate contact between the field operator and the laboratory personnel. Therefore, the field operator(s) must include on this folder all the information necessary for the analysis of the filter as well as information on any conditions or circumstances that might invalidate the sample or cause it to deviate from the normal. Figure 8 shows an exposed filter, filter folder with recorded data, and the recorder chart. The following information must be recorded on the folder by the indicated individuals. In some cases a separate data sheet is used for recording the data allowing the filter folder to be reused several times.

A. Operator Who Starts the Sampler

1. Filter number
2. Station number
3. Sampler number
4. Starting time
5. Initial flow rate (if using rotameter)
6. Date and initials
7. Summary of any unusual conditions that may affect results (e.g., subjective evaluation of the pollution that day, construction activity, meteorology, etc.)

B. Operator Who Removes Sample

1. Stop time, and if available, elapsed time.
2. Final flow rate (or flow-rate chart must accompany the sample)
3. Date and initials
4. Summary of existing conditions that may affect results (see A7 above, and 3 and 4 of Step 10)



Figure 8: Properly Prepared Filter Folder and
Accompanying Filter Mat with Recorder
Chart

C. Person Who Transfers Sample to Laboratory (if not done by person who takes sample)

1. Receiving date and initials
2. Shipping date and initials

SAMPLE ANALYSIS

Step 12. Sample Equilibration

The exposed filter is placed in the conditioning environment for 24 hours (see Section 2.9 for a description of conditioning chamber and environment). The 24-hour equilibration period should be adhered to (e.g., 20 to 28 hours) for uniformity of results. Many samples show a continued weight loss for several days, thus, unequal conditioning periods induce errors when data from different tests are compared.

If the conditioning environment is an air-conditioned room where filter preparation, weighing, and conditioning activities are performed, a hygrometer should be maintained out of the air-conditioning draft but in a location which receives good circulation. The relative humidity must be checked daily when exposed filters are being conditioned. Any relative humidity less than 50 percent and constant to within ± 5 percent is satisfactory. Temperature should be maintained to $\pm 3^{\circ}\text{C}$ at any level comfortable to the persons working in the room. It is important that relative humidity and temperature be the same for sample equilibration and filter equilibration (Step 4). Thus, they both should be done at the same facility or at facilities maintained at the same conditions.

If the conditioning environment is a desiccating chamber, a desiccating agent such as indicating activated alumina should be used. The desiccant should be checked daily and replaced when necessary as indicated by a color change in the desiccant.

Care should be exercised when placing filters in the desiccator or conditioning environment to make sure that the filter does not come in contact with loose dirt particles or the desiccant which might adhere to the filter and be weighed. Also, the filter should not be placed in a position such that some of the sample might fall or be knocked loose.

Also, when removing the exposed filter from the mailing envelope or glassine envelope, check to see if any of the collected particulates have come loose and are in the envelope. Recover as much of the particulates as possible using a small camels' hair brush to brush out the envelope.

Gnats and/or other bugs embedded in the particulates are removed with teflon-tipped tweezers, being careful not to displace any more of the particulate matter than necessary. If the number is excessive, greater than 10, report it to the supervisor for a decision on whether to accept or reject the sample.

Step 13. Gravimetric Analysis

Perform a balance check as specified in Step 5. Weigh exposed filters to the nearest milligram on the analytical balance. Record filter weights in the laboratory log book.

The weighing area should be in the conditioning environment if possible, otherwise the analytical balances should be as close as possible to the conditioning chamber in an area that is relatively free of air currents and maintained at the same temperature as the chamber. The filter must be weighed immediately, certainly no more than 5 minutes, after removal from the conditioning environment.

DATA PROCESSING

Step 14. Documentation and Sample Verification

The exposed filter weight is recorded in the laboratory log book and on the filter folder.

At this point all documentation is checked and compared for completeness and accuracy. The filter number on the filter, filter folder, and flow-rate chart (if included) should be the same and match the one in the laboratory log book. All data necessary for computing the concentration must be recorded on the filter folder as well as information on sampling date and location. The sample is voided if the filter numbers don't match or if any of the other pertinent data are missing.

The exposed filter is also inspected once again for signs of air leakage or physical damage to the filter that the operator may have overlooked but that could still invalidate the sample. Also, flow-rate values, environmental conditions, and operator remarks should be checked before the sample is declared valid.

Step 15. Calculations

Calculate the volume of air sampled and the mass concentration of suspended particulates as instructed in Sections 9.2 and 9.3 of Appendix A, respectively. Note that the equation for volume of air sampled in 9.2.2 of Appendix A is in error. It should be

$$V = \frac{Q_i + Q_f}{2} \times T.$$

For samplers equipped with a flow-rate recorder the calculations are performed as described in Addenda A of Appendix A.

Step 16. Document and Report Data

Daily concentration levels with required identifying information are recorded in micrograms per cubic meter on the SAROAD Daily Data Form. See Users Manual: SAROAD (Storage and Retrieval of Aerometric Data), APTD-0663, for detailed instructions for accomplishing this. The original calculations should be filed in the laboratory log book.

2.2 Flow Rate Calibration

A. Calibration of Orifice Unit

The orifice calibration unit with different resistance plates, as shown in Figure B3 of Appendix A, is the specified unit for calibrating the flow rate of both rotameter and flow-rate recorder equipped samplers. However, this orifice calibration unit itself must first be calibrated against a positive displacement primary standard.

Directions for calibrating the orifice calibration unit against the primary standard are given in Section 8.1.1 of Appendix A.

The orifice calibration unit should remain virtually unchanged over a period of several years under normal use. Its calibration against a standard serves primarily as a check for changes due to some form of physical damage.

The orifice calibration unit should be calibrated with a primary standard when it is first purchased. A deviation of more than ± 4 percent at any point from the average calibration curve furnished by the manufacturer probably means that the orifice has been damaged in shipment and should not be accepted (Ref. 1).

Orifice units in use should be visually inspected for visible signs of damage to the orifice before each use. A calibration check should be made anytime the unit, especially the orifice itself, appears to have any nicks or dents.

Calibration checks against a primary standard should be made once a year for all orifice units. The manufacturer's average calibration curve should continue to be used unless the new calibration deviates from it by more than ± 4 percent at any one point along the curve. When deviations from the manufacturer's average calibration curve are larger than ± 4 percent and there are no visible signs of damage to the orifice, the calibration should be repeated by another operator. If the large deviations persist (after the primary standard has been checked and found satisfactory) a new average calibration curve is constructed using the results from at least five sets of calibration data.

B. Sampler Calibration

Samplers must be calibrated when first purchased, after major maintenance on the sampler (e.g., replacement of motor or motor brushes), any time the flow-rate measuring device (i.e., rotameter or recorder) has to be replaced or repaired, or any time a one-point calibration check (see Section 2.6 for a description of this check) deviates more than ± 6 percent from the calibration curve.

It is expected that samplers will have to be returned to the laboratory for routine maintenance and calibration after 25 to 30 operating days. This is based on the average brush life of a sampler operating on 100 volts. Samplers operating on line voltage (120 volts) will require brush replacement and thus calibration more often.

Calibrations performed in the laboratory must be corrected or repeated on site for samplers operating at stations where ambient barometric pressure or temperature is significantly different from those in the laboratory.

The orifice calibration unit with a set of resistance plates is used to calibrate either or both the rotameter and recorder equipped high volume samplers in the field or in the laboratory.

Figure 9 shows the apparatus required for the calibration of a high volume sampler in the field. The apparatus was arranged in this manner for illustration purposes only. In actual practice it is recommended that the sampler and recorder be left in the shelter while calibrating. Specifically, care should be taken not to restrict the air flow into the orifice unit or out of the motor unit. The calibration setup for the rotameter equipped sampler is exactly the same with the exception that a rotameter replaces the flow rate recorder in Figure 9.

In using the orifice calibration unit to calibrate a sampler, corrections must be made to the indicated flow rate if the ambient barometric pressure or temperature is substantially different from the pressure or temperature values recorded when the orifice unit was calibrated. Calculate the corrected flow rate as follows:

$$Q_2 = Q_1 \left[\frac{T_2 P_1}{T_1 P_2} \right]^{1/2}$$

where

Q_2 = corrected flow rate, m^3/min ;

Q_1 = uncorrected flow rate read from the orifice unit calibration curve for a given pressure in inches of water;

T_1 = absolute temperature when orifice unit was calibrated, $^{\circ}K$;

P_1 = barometric pressure when orifice unit was calibrated, $mmHg$;

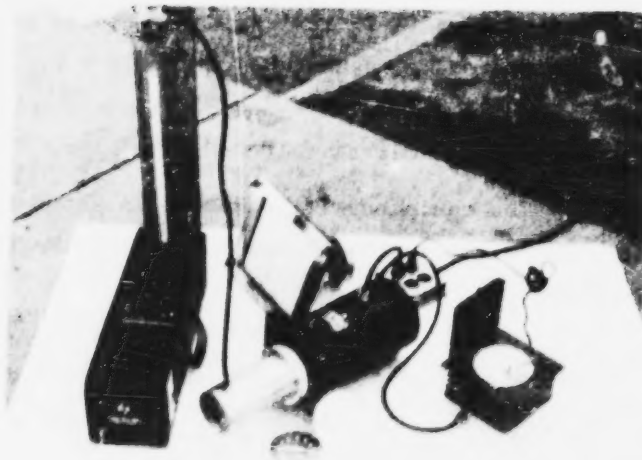


Figure 9: Typical Field Calibration Setup
for Modified High Volume Sampler

T_2 = absolute temperature while calibrating the sampler, same units as T_1 , and

P_2 = barometric pressure while calibrating sampler, same units as P_1 .

For a given pressure (i.e., $P_1 = P_2$) Figure 10 shows the percentage change of Q versus temperature differences. If T_2 is greater than T_1 , the percentage change is positive; if T_2 is less than T_1 , the percentage change is negative. The same procedure is used to correct for pressure differences.

1. Sampler Equipped with Rotameter

Equipment Setup - The equipment is connected as shown in Figure 9, with the exception that a rotameter is used instead of the pressure transducer and recorder.

- 1) Replace the filter adapter with the orifice calibration using the resistance plate with 18 holes (seventeen in a circle and one in the center of the plate) to approximate the resistance of a clean filter.
- 2) Connect the rotameter to pressure tap at exhaust end of high volume motor with a section of tubing (rotameter just replaces the recorder in Figure 9). This is a positive pressure, so connection is made at the bottom of the rotameter. The rotameter and tubing used in calibration must be used when making flow-rate readings in the field.
- 3) Connect the manometer to the orifice calibration unit. Caution: The orifice unit exerts a negative or vacuum pressure. The manometer end not connected to the orifice unit must be open to the atmosphere.

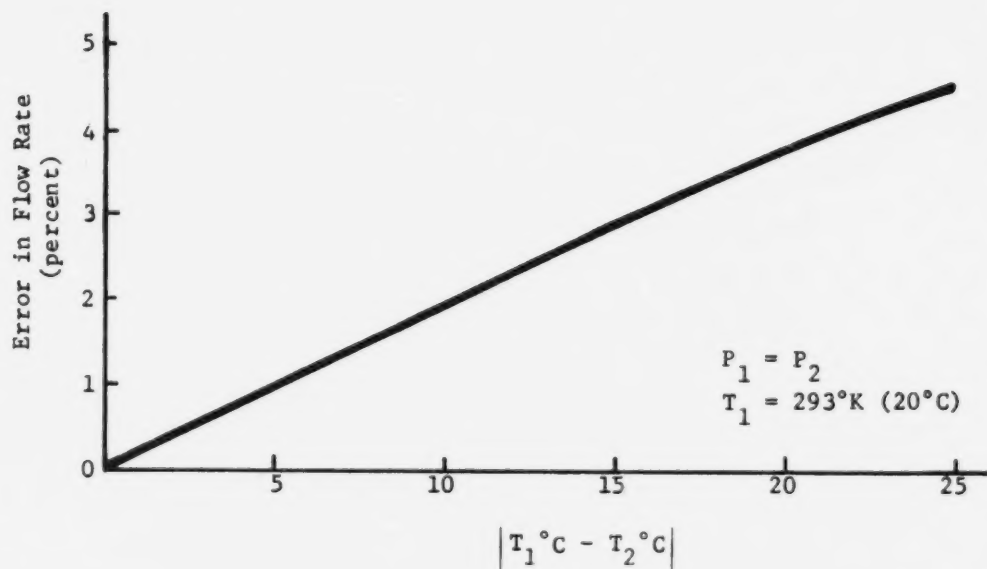


Figure 10: Percent Change in Flow Rate Versus Temperature Variations (Orifice Calibration Unit)

Calibration Procedure

- 1) Plug sampler into 120 volt source, while checking manometer to insure that the orifice pressure drop does not exceed the range of the manometer. Let the sampler run for about 5 minutes.
- 2) Read the manometer pressure in inches of water, record on calibration data sheet (Figure 11). Convert to flow rate using the orifice unit calibration chart (correct for temperature and pressure using the equation on page 26 if needed) and record in column 3 of the calibration data sheet.
- 3) Set the rotameter reading (wide part of float) as near as possible to the correct flow rate, as determined in 2 above (if the rotameter has arbitrary units, set to the normal flow rate expected with a clean filter), by adjusting the brass hexagonal nut at the top of the rotameter. Once adjusted, tighten the lock nut and seal to prevent the setting from changing.
- 4) Record the rotameter reading in Column 4 of the calibration data sheet.
- 5) Replace the resistance plate in the orifice unit with the one with the next fewer number of holes.
- 6) Turn on the sampler and record on the calibration data sheet the manometer pressure in inches of water, the corrected flow rate from the calibration chart, and the rotameter reading.
- 7) Repeat Steps 5 and 6 for the remaining resistance plates getting a total of 5 or 6 different flow rates.
- 8) On graph paper, plot rotameter readings (Column 4) versus flow rate in m^3/min (Column 3).
- 9) Construct a best-fit, smooth curve to the 5 or 6 points by eye or by a curve fitting technique such as a least squares fit.

CALIBRATION DATA SHEET

Orifice Unit No. _____ Initials _____

Sampler No. _____ Date _____

Indicator No. _____ Barometer _____ mmHg
(Rotameter/Recorder)

Temperature _____ °C

Run Number 1	Manometer in. Water 2	Actual (Corrected) Flow Rate m ³ /min 3	Indicator Reading 4

Figure 11: Sample Calibration Sheet

- 10) Recheck any point that deviates more than ± 5 percent from the smooth, best-fit curve. Calculate the percent deviation by taking the flow rate of the point in question as Q_o and the flow rate from the calibration curve as Q_c for the same rotameter reading and compute

$$\text{percent deviation} = \frac{Q_o - Q_c}{Q_c} \times 100.$$

Replot the point as the average of the two values.

2. Sampler Equipped with Transducer and Recorder

Equipment Setup - The equipment is connected as illustrated in Figure 9 (see page 26 for proper cautions to take in setting up equipment).

- 1) Replace the filter adapter with the orifice calibration unit using the 18-hole resistance plate to simulate a clean filter.
- 2) Connect one leg of the manometer to the orifice calibration unit and vent the other leg to the atmosphere.
- 3) Install a clean recorder chart and check the recorder for proper operation. Zero the pen if necessary (see 2b of Step 9).

Calibration Procedure

- 1) Connect the sampler directly to a 120 V source, bypassing the step-down transformer if it is normally used. Let the sampler run for about 5 minutes.
- 2) Read the differential pressure as indicated by the manometer and record the reading in Column 2 of the calibration data sheet (Figure 11). Convert to flow rate in m^3/min using the orifice unit calibration chart (using the correction for temperature and pressure if applicable) and record in Column 3.

- 3) Adjust the span of the recorder so that the recorder pen is indicating the correct flow rate (if the recorder chart is in arbitrary units, set to the normal flow rate expected with a clean filter).
- 4) Shut the sampler off. Check zero and reset if necessary. If it is necessary to make a zero adjustment, then Steps 2, 3 and 4 are repeated until no span or zero adjustments are required. Record the recorder chart deflection in Column 4 of the calibration data sheet.
- 5) Change the resistance plate now in the orifice calibration unit to the one with the next fewer number of holes.
- 6) Turn on the sampler and convert the differential pressure as given by the manometer to the corrected flow rate.
- 7) Record the manometer pressure in inches of water, the actual corrected flow rate from the calibration chart in m^3/min , and the recorder deflection on the calibration data sheet as shown in Figure 11.
- 8) Repeat Steps 5, 6 and 7 for the remaining resistance plates getting a total of five or six different flow rates.
- 9) Plot on graph paper the recorder deflection (Column 4) versus flow rate in m^3/min (Column 3).
- 10) Construct a best-fit, smooth curve through the 5 or 6 points by eye or by a curve fitting technique such as a least squares fit.
- 11) Recheck any point that deviates more than ± 5 percent from the smooth curve. Calculate the percent deviation by taking the flow rate of the point in question as Q_o and the flow rate from the calibration curve as Q_c for the same recorder deflection and compute

$$\text{percent deviation} = \frac{Q_o - Q_c}{Q_c} \times 100.$$

2.3 Relative Humidity Indicator Calibration

The relative humidity indicator used for monitoring the conditioning environment should be checked against a wet-bulb, dry-bulb psychrometer or equivalent every six months. At least a two-point calibration should be made by comparing readings made in the conditioning environment and then moving the relative humidity indicator outdoors or perhaps just out of the conditioning room for a second comparison. If the indicator readings are within ± 6 percent of the psychrometer values, continue to use the relative humidity indicator. If they disagree by more than ± 6 percent, either have the indicator calibrated or purchase a new one.

2.4 Analytical Balance Calibration

The balance calibration should be verified when the balance is first purchased, any time the balance has been moved or subjected to rough handling, or when a standard weight cannot be weighed within ± 0.5 mg of its stated weight. Weighing a set of at least 5 standard weights, covering the weight range normally encountered in weighing filters, can serve as a verification. If at any time one or more of the standard weights cannot be measured within ± 0.5 mg of its stated value, have the balance recalibrated. The manufacturer should perform the calibration and subsequent adjustments.

2.5 Elapsed Time Indicator Check

The elapsed time indicator should be checked every six months against a time piece of known accuracy over a 24-hour period. This could be accomplished on site or in the laboratory. If the indicator shows any signs of being temperature sensitive, it should be checked on site during each season of the year.

A gain or loss of more than 4 minutes in a 24-hour period warrants an adjustment or replacement of the indicator.

2.6 Special Checks for Auditing Purposes

In making special checks for auditing purposes it is important that all checks be performed without any special preparation or adjustment of the system (see Section 3.1 for further discussion). It is felt that when first starting a quality assurance program, seven special checks are required to properly assess data quality. The necessity of continued performance of each check can be evaluated as auditing data become available. A checking or auditing level of 7 checks out of 100 sampling periods is used here for illustration purposes. The supervisor will specify the auditing level to be used according to monitoring requirements.

For the case where one sample is collected every sixth day, an auditing level of 1 check per month is recommended. This would result in an auditing level of approximately 3 checks ($n = 3$) for a lot size of 15 ($N = 15$) for data reported quarterly. Directions for performing each of the checks are given here. Proper use of the resulting data along with desirable control limits is given in Section 3.1 of the Supervision Manual.

A. Analysis of Filter Surface Alkalinity

It is recommended that filter surface alkalinity (pH) be audited at the beginning of a quality assurance program. It is further recommended that only filters with a pH between 6.5 and 7.5 be used. If auditing results show that the manufacturer can consistently supply filters that are within an acceptable pH range, this check may be discontinued. Perform the check in the following manner.

- 1) Randomly select 7 filters out of every 100 filters.
- 2) Remove a small sample (e.g., a 3" x 3" square) from one filter. Place the sample in a small beaker and cover with 15 ml deionized water. Bring to a slow boil for 1 minute. Cool to room temperature. Measure the pH with a pH meter. If a pH meter is not available, use fresh indicating litmus paper, such as Fisher Scientific Short Range Alkacid Test Papers.

- 3) Record the 7 measured pH values and forward to the supervisor. This check should be made as part of Step I in Figure 1. The supervisor will reject the lot if a pH outside the range of 6.5 to 7.5 is measured.

B. Weighing Checks

Weighing checks are made as soon as practical before or after the regular weighing. No more than 30 minutes should elapse between weighings of exposed filters when the weighing is carried out in the conditioning environment and even less if the filters are removed from the conditioning environment. Weighing checks are performed as part of Steps 5 and 13 in Figure 1, pages 4 and 5.

The check must be independent, i.e., performed by a person other than the one doing the regular weighings. Treat these as go/no-go checks, i.e., if one check exceeds the control limits, reweigh all filters and use the check values as the correct ones. If, however, no check exceeds the limits, the check values are recorded but no changes are made in the original weights.

Clean Filters - Clean filters are normally weighed in batches. This allows for the sampling to be performed and corrections to be made before the filters are used.

- 1) Divide into lot sizes of 100 or less and weigh.
- 2) Randomly select and reweigh 7 filters from each lot of 100.
- 3) If any one of the 7 check weights differs more than 1.0 mg from the original weight, reweigh all the filters in that lot.
- 4) Record both weights in the laboratory log book with the filter number. Use the weight determined by the check as the correct one. The lot is accepted with no changes made if all checks differed from the original weights by less than 1.0 mg.

Exposed Filters - Due to the necessity of weighing exposed filters immediately after a 24-hour conditioning period, it may be impossible to have lot sizes greater than 10 or 20. In order to allow for corrections to the lot, it is necessary to perform the audit as the filters are weighed, regardless of the size of the lot.

- 1) Randomly select and reweigh 4 out of every lot size of 50 or less (this would mean 100% checking if 4 or less exposed filters are weighed at one time). If lot sizes of 50 or greater are possible, reweigh 7 from each lot.
- 2) Reweigh all filters in a lot if any check differs by more than ± 2.7 mg (assuming $\sigma = 0.9$ mg) from the original weight.
- 3) Accept the lot with no change if all checks are within ± 2.7 mg of the originals.
- 4) Record original and check weights in the laboratory log book.

C. Flow-Rate Check

Flow-rate checks should be independent and random; that is, a person other than the regular operator makes the check. Also, the regular operator should not know in advance when the check is to be made. The check is made in the following manner.

- 1) As part of the routine operations the regular operator services the sampler and measures the initial flow rate, Q_1 , as directed in A of Step 9, page 11.
- 2) Make an independent measurement within 15 minutes or less of the operator's measurement. Record the check value, Q_1' , in the site log book.
- 3) Make an additional flow-rate measurement, Q_m within ± 1 hour of the midpoint of the sampling period. Record the value in the site log book.

- 4) Within 15 minutes or less of the regular operator's final flow-rate measurement, make an independent check and record the value in the site log book as Q_f' .
- 5) The regular operator makes a final flow-rate measurement, Q_f , and records it in the site log book.
- 6) Values of Q_1 , Q_1' , Q_m' , Q_f , and Q_f' are reported to the supervisor.

D. Calibration Check

Independent calibration checks should be made on site. Portable calibration equipment as shown in Figure 9, page 27 is used. Perform calibration checks according to the following procedure.

- 1) Set up equipment.
- 2) Select one of the resistance plates and obtain the actual flow rate, Q_a , and the rotameter reading, following the calibration procedures given on page 30, Section 2.2.
- 3) Convert rotameter reading to flow rate, Q_r , using the calibration curve and making corrections for ambient temperature and pressure.
- 4) Compute

$$\text{percent difference} = \frac{Q_r - Q_a}{Q_a} \times 100.$$

- 5) Report the percent difference to the supervisor.
- 6) If the percent difference is as large as 6, a complete calibration should be performed before sampling is resumed.

E. Elapsed Time Between Collection and Analysis

Elapsed time between sample collection and analysis is important in estimating error due to loss of weight of sample particulates having high organic matter content.

The recommended minimum auditing level is 7 checks ($n=7$) out of every 100 samples ($N=100$) for networks generating 100 or more samples per quarter, and $n=3$, $N=15$ where sampling occurs every sixth day.

Perform the check by randomly selecting the samples to be checked. From the data sheet, obtain the end time and date of the sampling period, and obtain the time and date of the weighing of the exposed filter from the filter processing data log book.

Determine the elapsed time in days, and subtract 1 for the conditioning period. Report this value to the supervisor.

F. Data Processing Check

In auditing data processing procedures, it is convenient and allows for corrections to be made immediately if checks are made soon after the original calculations have been performed. In particular, this allows for possible retrieval of additional explanatory data from field personnel when necessary. For networks generating as many as 100 samples per quarter, the recommended auditing level of 7 checks ($n=7$) for a lot size of 100 ($N=100$) can be followed. Networks consisting of one or two samplers operating every sixth day, the minimum level of 4 checks ($n=4$) for all lot sizes less than 50 ($N<50$) should be used.

The check must be independent; that is, performed by an individual other than the one who originally reduced the data. The check is made starting with the raw data on the data sheet or flow-rate recorder chart and continuing through recording the concentration in $\mu\text{g}/\text{m}^3$ on the SAROAD form.

If the mass concentration of suspended particulates computed by the check, S.P._c , differs from the original value, S.P._o , by as much as ± 3 percent, all samples in that lot are checked and corrected. The check value is always given as the correct value.

Check values are recorded in the data log book and reported to the supervisor.

2.7 Special Checks to Detect and Identify Trouble

The following checks may be required when: 1) a quality assurance program is first initiated in order to identify potential problem areas, and 2) at any later time when it becomes increasingly difficult to meet the performance standards of the auditing program to identify and evaluate trouble areas. The required information is primarily a description of the sampling site ambient environment. Specific areas of required information are:

- 1) average concentration of acid gases,
- 2) average percent of organic matter present in collected particulates,
- 3) average particulate concentration,
- 4) average flow-rate change per 24-hour sampling period,
- 5) diurnal pattern of particulate matter, and
- 6) source voltage variation for 24-hour sampling period.

A. Average Concentration of Acid Gases

This information is not required if the ambient atmosphere is known to be free of such acid gases as SO_2 and NO_2 .

Values can be derived from previous measurements made on site or in the general site area. In many cases a good estimate can be made from a knowledge of the emission sources in the area. The primary requirement is to know, in general, whether or not there are acid gases in the ambient atmosphere. An absolute value is not required.

Record the measured/estimated concentration values of SO_2 and NO_2 on the form for site evaluation data as shown in Table 1.

Table 1: Sampling Site Evaluation Data

Parameter	Value
1. Average Concentration of Acid Gases	$\text{SO}_2(\mu\text{g}/\text{m}^3) =$, $\text{NO}_2(\mu\text{g}/\text{m}^3) =$
2. Average Percent of Organic Matter in Particulates	% O.M. =
3. Average Suspended Particulate Concentration	$\overline{\text{S.P.}}(\mu\text{g}/\text{m}^3) =$
4. Average Flow-Rate Change per 24 hours	$\overline{\Delta Q}(\text{m}^3/\text{min}) =$
5. Diurnal Particulate Pattern	(present as a graph)
6. Source Voltage Variation per 24 hours	(present as a graph)

B. Organic Matter as a Percent of Total Particulate Matter

An average value for organic content of particulate matter for a given site can be determined from previous measurements made on site or in the general area around the site. In order to obtain valid results, the collected particulates should be analyzed for organic matter immediately after the 24-hour conditioning period. The test is usually made in terms of benzene soluble organics. In some cases an estimate may be adequate if there is a good knowledge of the emission sources in the area.

Record the measured/estimated percentage on the form for site evaluation data as shown in Table 1.

C. Average Particulate Concentration

From previous data (e.g., from the previous year's data), obtain an average concentration level. If no previous data are available, use data from nearby sites and previous experience to make an estimate. If an estimate is used in the beginning, average each quarter's data and use that average until a year's data have been collected. Use the annual mean when available.

Record the measured/estimated value of particulate concentration on the form for site evaluation data as shown in Table 1.

D. Average Change in Flow Rate for a 24-Hour Sampling Period

Obtain the initial and final flow rates from at least 20 sampling periods. Compute

$$\Delta Q = Q_i - Q_f$$

for each period.

Compute the average flow rate change, $\overline{\Delta Q}$, by adding all the ΔQ 's and dividing their sum by the number of ΔQ 's used.

Record $\overline{\Delta Q}$ on the form for site evaluation data as shown in Table 1.

E. Diurnal Pattern of Particulate Matter

If $\overline{\Delta Q}$ from D above was less than $0.30 \text{ m}^3/\text{min}$ ($\sim 11 \text{ ft}^3/\text{min}$), this information is not needed. For cases where $\overline{\Delta Q} > 0.30 \text{ m}^3/\text{min}$, construct a graph of suspended particulate concentration versus time for a typical or average 24-hour sampling period for that site. Relative comparisons of the lowest and highest values and the approximate times of their occurrence during the sampling period are of importance. Data from measurements made with a tape sampler (e.g., hour or two hour averages), or other methods giving averages for time periods of 4 hours or less, can be used to construct the graph. In some areas a reasonable estimate of the diurnal pattern can be made from a knowledge of the operating cycle of the local emission sources.

Construct a graph from the measured/estimated values and attach to the form for site evaluation data as shown in Table 1.

F. Source Voltage Variation for 24-Hour Sampling Period

For a given sampling site and with the sampler operating, monitor the source voltage over the 24-hour sampling period. This can be done with a continuous recording device or an indicating voltmeter read and recorded every hour. This check should be performed on at least two different week days.

Plot the source voltage (hourly values) versus time (label and use strip chart record if used) and attach to the form for site evaluation data as shown in Table 1.

2.8 Maintenance

The three most frequently required maintenance actions include replacement of the sampler motor brushes, replacement of the faceplate gasket, and cleaning of the rotameter.

A. Sampler Motor

Motor brushes usually require replacement after 400 to 500 hours of operation at normal line voltage (115V). The brushes should be replaced before they are worn to the point that motor damage can occur. The optimum replacement interval must be determined from experience. Manufacturer's instructions should be followed in replacing the brushes.

B. Faceplate Gasket

A worn faceplate gasket is characterized by a gradual blending of the interface between collected particulates and the clean filter border. Any decrease in the original sharpness of this interface indicates the need for a new faceplate gasket.

The old gasket can be removed with a knife and the surface properly cleaned. A new gasket is then sealed to the faceplate with rubber cement or double-sided adhesive tape.

C. Rotameter

Small particles may become lodged in the air cavity of the rotameter resulting in erratic behavior of the float. Alcohol is a safe fluid to use for cleaning the rotameter. The rotameter should be cleaned and calibrated at any sign of foreign particles or moisture deposits in the air column or erratic behavior of the float. Also, the rotameter should be cleaned prior to routine calibration. The rotameter is discarded if any physical damage such as a crack in the plastic sleeve is observed.

2.9 Facility and Apparatus Requirements

A. Facility

Primary facilities required in High Volume sampling are a central laboratory and individual sampling stations. The laboratory should be equipped for filter/sample processing and for calibrations and maintenance.

1. Filter Conditioning and Weighing Area

Ideally the filter conditioning area would be a room large enough to accommodate filter processing, equilibration, weighing operations, and filter library. The room would be equipped with the necessary air conditioning equipment to maintain a preset temperature and relative humidity. Also, relative humidity and temperature measurement instruments are required.

In the event that room is not available, a desiccating chamber, such as a converted oven, refrigerator, incubator or a commercially manufactured chamber equipped with trays for holding desiccant and v-shaped racks approximately 4" high for holding filters may be used. The weighing area should be located next to the conditioning chamber in an area that is relatively free of air currents.

In all cases the conditioning environment should be free of acidic or basic gases that may react with the filter media or the collected particulates during filter/sample conditioning.

2. Calibration Area

To help insure a minimum of calibration error, a permanent calibration area should be established in the laboratory. The area should be equipped with an orifice calibration unit, a differential manometer, and a positive displacement meter. Temperature and barometric pressure indicators should be available also.

3. Maintenance Area

A sufficiently large area should be designated as the maintenance and test area. It should be equipped with the tools required for routine sampler maintenance, such as brush or motor replacement, and auxiliary equipment maintenance, such as the adjustment and repair of pressure transducer and flow-rate recorders.

B. Apparatus

Specifications for the apparatus are given in Section 5 of Appendix A. Table 3 is a listing of the apparatus with approximate costs. Costs are computed for placing a sampler (standard and modified) on site complete for sampling, and for the laboratory equipment, which would be prorated across several sampling stations.

Certain items of equipment listed as additional sampler equipment are not required in the reference method, but if used could increase data quality.

A filter paper cartridge provides a means for allowing the filter changes to be made in the laboratory and provides protection for the clean and/or exposed filter during transit to or from the sampling site. The cartridge reduces the risk of loss of sample or otherwise invalidation of a sample when changing filters during adverse weather conditions.

The 7-day timer and elapsed time indicator allow one to service the sampler at his convenience and to have the sampler operate at some preset time by setting the 7-day timer. An accurate measurement of the sampling time is given by the elapsed time indicator.

In special situations it may be desirable to maintain as nearly as possible a constant flow rate. Constant flow regulators have been developed which maintain the flow rate to within 10 percent of its initial value. In certain situations when a flow regulator is not available, but the flow rate is known to vary due to variations in the power line voltage, a constant voltage regulator can be used between the voltage source and the sampler to maintain a constant source voltage.

Paper supplies (not listed in Table 3) required in the High Volume Method include manila folders (see Figure 8), glassine envelopes to protect the sample against absorption of moisture during transit, and suitable mailing envelopes large enough to accept the folded filter and filter folder and small enough to hold them firmly so that the filter cannot move around relative to the folder.

In addition to the above paper supplies, three record books suitable for use as a laboratory data log book, a calibration log book, and a maintenance log book must be purchased.

Table 2: Apparatus Used in the High-Volume Method

Item of Equipment	Approx. Cost 1972	Associated Error	Standard Sampler	Modified Sampler
1. Standard Shelter	\$ 56		✓	✓
2. Sampler (Less Filter Holder)	85		✓	✓
3. Additional Sampler Equipment				
a) 8" x 10" stainless steel filter holder	28		✓	✓
b) Filter paper cartridge [need 2/sampler]	34*	Loss of Sample		
c) 7 day timer	39*			
d) Elapsed time indicator	30*	Time		
e) Constant flow regulator	150*	Flow rate		
f) Constant voltage regulator	270*	Flow rate		
g) Step-down transformer	26		✓	✓
h) Pressure transducer & continuous flow rate recorder	94	Flow rate		✓
i) Rotameter	<u>9</u>		✓	
COST OF SAMPLER ON SITE			\$ 204	\$ 289
4. Calibration				
a) Positive displacement meter (std)	1,000			
b) Orifice calibration unit	74			
c) Barometer & thermometer	100			
5. Filter Conditioning Envir.				
a) Conditioning room or desiccator	1,000 or 300			
6. Weighing				
a) Balance	850			
b) Air pollution weighing chamber	230			
7. Filter Preparation				
a) Light source	30			
b) Numbering device	<u>20</u>			
LABORATORY EQUIPMENT COST	\$ 4,125 or \$3,425			

* Not computed in cost

PART II. SUPERVISION MANUAL

3.0 GENERAL

Consistent with the realization of the objectives of a quality assurance program as given in Section 1.0, this manual provides the supervisor with brief guidelines and directions for:

- 1) the collection and analysis of information necessary for the assessment of high volume data quality,
- 2) isolating, evaluating, and monitoring major components of system error,
- 3) changing the physical system to achieve a desired level of data quality,
- 4) varying the auditing or checking level to achieve a desired level of confidence in the validity of the outgoing data, and
- 5) selecting monitoring strategies in terms of data quality and cost for specific monitoring requirements.

This manual provides brief directions that cannot cover all situations. For somewhat more background information on quality assurance see the Management Manual of this document. Additional information pertaining to the High Volume Method can be obtained from the final report for this contract and from the literature referenced at the end of the Management Manual.

Directions are written in terms of a 24-hour sampling period and an auditing level of $n=7$ checks out of a lot size of $N=100$ for illustration purposes. Special instructions for auditing operations where sampling is performed every sixth day are given also. Information on additional auditing levels is given in the Management Manual.

Specific actions and operations required of the supervisor in implementing and maintaining a quality assurance program as discussed in this Manual are summarized in the following listing.

- 1) Data Assessment
 - a) Set up and maintain an auditing schedule.
 - b) Qualify audit results (i.e., insure that checks are independent and valid).
 - c) Perform necessary calculations and compare with suggested performance standards.
 - d) Make corrections or alter operations when standards are exceeded.
 - e) Forward acceptable qualified data, with audit results attached, for additional internal review or to user.
- 2) Routine Operation
 - a) Obtain from the operator immediate reports of suspicious data or malfunctions. Initiate corrective action or, if necessary, specify special checks to determine the trouble; then take corrective action.
 - b) On a daily basis, evaluate and dispose of (i.e., accept or reject) data that have been identified as questionable by the operator.
 - c) Examine operator's log books periodically for completeness and adherence to operating procedures.
 - d) Approve filter processing data sheets, calibration data, etc., for filing by operator.
 - e) File auditing results.
- 3) Evaluation of Operations
 - a) Evaluate available alternative monitoring strategies in light of your experience and needs.
 - b) Evaluate operator training/instructional needs for your specific operation.

3.1 Assessment of High Volume Data

Procedures for implementing and maintaining an auditing program to assess data quality are presented in this section. Two auditing programs are discussed. The first and preferred program involves auditing individual variables. The second program consists of auditing the entire measuring process by comparing the final results from the field sampler to the results obtained with a reference sampler. This second method is presented here as an alternative to be used in situations where implementation of the first program is impossible or impractical.

Throughout this discussion and the rest of this document, the term "lot" is used to represent a set or collection of objects (e.g., measurements or observations), and the "lot size" designated as N is the number of objects in the lot. The number of objects in the lot to be tested or measured is called the "sample size" and is designated by n . The term "auditing level," used interchangeably with "checking level," is fully described by giving the sample size, n , and the lot size, N .

3.1.1 Assessment by Auditing Individual Variables

A valid assessment of a lot of high volume data can be made at a given level of confidence with information derived from special checks. Figure 12 summarizes the quality control checks applied at various check points in the measuring process. Each check or operation is represented by a box. The numbers at the top left hand side of each box identify the step in the process, as given in Figure 1 of the Operations Manual, at which the check is performed.

Boxes enclosed by heavy lines represent 100 percent sampling; i.e., these checks will be performed for each filter passing through the system. All other checks are to be performed at the prescribed auditing level. All but three of the checks are treated on a go/no-go basis. That is, a standard is defined and the lot or individual item is accepted or rejected on the basis of the check results. Certain rejected lots are corrigible, i.e., they are capable of being corrected. Specifically, lots rejected because of weighing or data processing errors are accepted after the errors have been located and corrected.

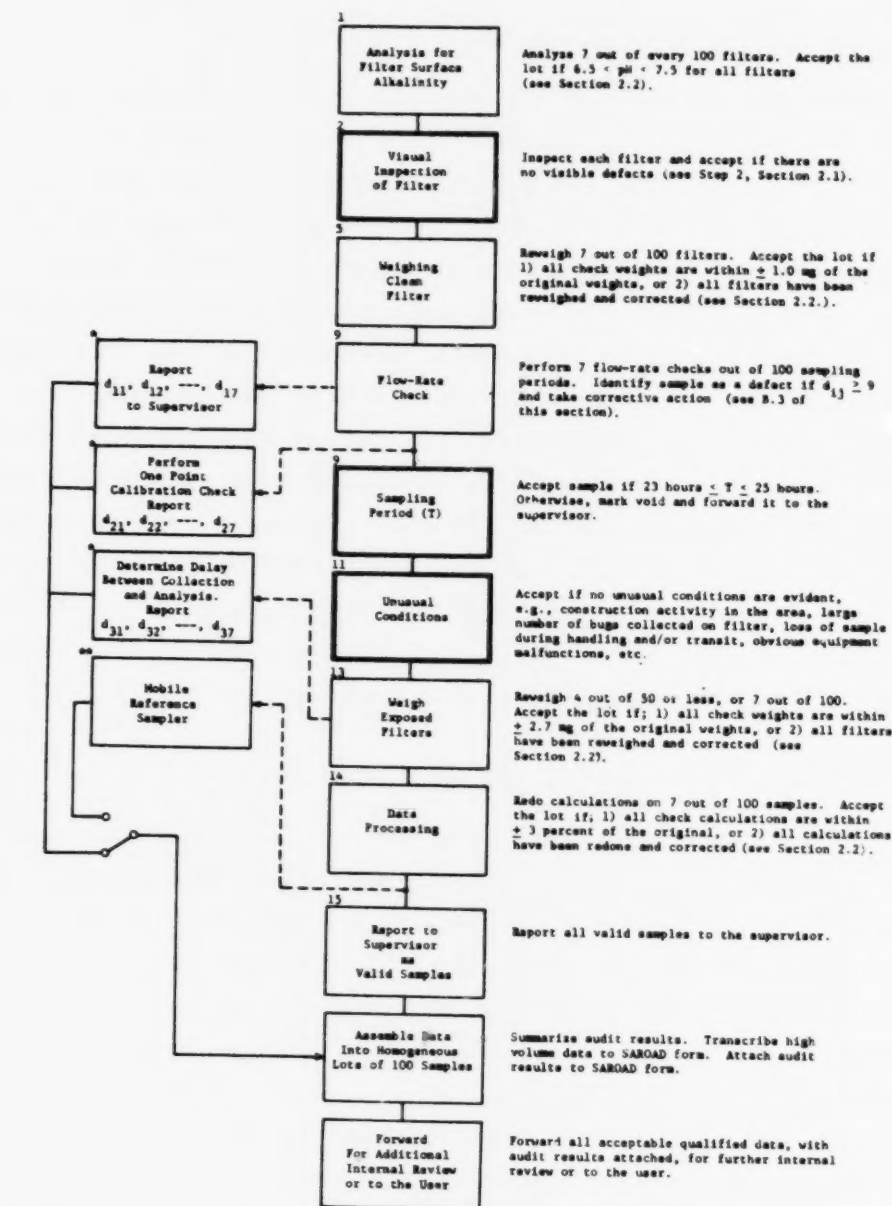


Figure 12: Flow Chart of Quality Control Checks in the Auditing Program

The three checks not treated on a go/no-go basis are; 1) flow-rate check, 2) calibration check, and 3) a check of elapsed time between collection and analysis. These checks are performed at the prescribed auditing level. Action for correcting system deficiencies can be taken as the result of any one check, however, there is usually no clear-cut way of correcting previous data. Therefore, results of these three checks are reported and used in assessing data quality as described in Section 4.1 of the Management Manual.

A. Required Information

The seven checks to be performed at the prescribed auditing rate are:

- 1) filter surface alkalinity,
- 2) weighing of clean filters,
- 3) flow-rate check,
- 4) calibration check,
- 5) weighing of exposed filters,
- 6) elapsed time interval between sample collection and analysis, and
- 7) data processing check.

Auditing Checks 2, 4, 5 and 7 are required for all monitoring situations while certain conditions may eliminate the need for one or more of Checks 1, 3, and 6.

It is not necessary to audit the filter surface alkalinity (Check 1) if the manufacturer has performed control checks during the manufacturing process and certifies that the filter pH is between 6.5 and 7.5. Otherwise, it is recommended that this audit be performed.

The flow-rate check (Check 3) as described in Section 2.6 of the Operations Manual is not required when the sampler is equipped with a continuous flow-rate recorder.

A check of the elapsed time between collection and analysis (Check 6) is not required of networks in which the operator delivers the exposed filter to the laboratory for conditioning and analysis within less than

24 hours of the sampling period end time or for sampling sites where the organic content is less than 10 percent of total particulates. It is recommended that the audit be performed for any situation in which the sample is mailed to the laboratory for analysis and organic matter constitutes more than 10 percent of total particulates by weight.

Directions for performing the above 7 listed checks are given in the Operations Manual, Section 2.6. Directions for insuring independence and proper randomization in the auditing process and for the evaluation of the results are presented in this section.

B. Collection of Required Information

1. Filter Surface Alkalinity

This check can be performed by the operator or any individual capable of following the procedures given in A of Section 2.6 in the Operations Manual. If the pH range of 6.5 to 7.5, or any other specified range, is to be adhered to, the lot is rejected anytime a pH is measured outside the range (see A of Section 2.6 concerning rejecting lots) or accepted as good after 7 filters have been analyzed and all pH values are within the prescribed range.

Report the limits of the acceptable range and the auditing level.

2. Weighing Clean Filters

The weighing check should be independent, i.e., performed by someone other than the person performing the original weighings. Directions for randomly selecting the 7 filters for reweighing and performing the check are given in B of Section 2.6.

The lot is accepted as good if 1) all check weights are within ± 1.0 mg of the original weights or 2) all filters have been reweighed.

Report the standard (e.g., ± 1 mg) used to judge the weighing process, and the auditing level, on the form in Figure 13 of C below.

3. Flow-Rate Check

Procedure for Performing the Check - Samples from individual sites should be combined into lots. For sites where 50 or more samples are collected each quarter, a minimum of 7 randomly spaced checks per quarter is recommended. A minimum of 3 checks per quarter is recommended for sites operating every sixth day, thereby generating 15 or less samples a quarter.

Randomly select 7 sampling periods from the coming quarter for sites where the lot size is expected to be as large as 50. Record dates. The operator should not be aware of when the checks are to be performed.

For sites where the lot size is 15 or less, randomly select 1 sampling period each month. Record these dates and perform flow-rate checks as scheduled.

Directions for performing the check are given in C of Section 2.6.

Treatment of Data - Obtain from the operator values of Q_i , Q_i' , Q_m' , Q_f , and Q_f' as described in Section 2.6.

Calculate the average flow rate using Q_i and Q_f as measured by the operator by

$$\overline{\Delta Q_j} = \frac{Q_{ij} + Q_{fj}}{2}$$

where j is the j^{th} check performed during the auditing period.

Calculate the average flow rate using the check values Q_i' , Q_m' , and Q_c' by

$$\overline{\Delta Q_j'} = \frac{Q_i' + 4Q_m' + Q_f'}{6}.$$

Note that if the measurement Q_m' was not measured within ± 1 hour of the true midpoint of sampling period, $\overline{\Delta Q_j'}$ should be computed by the following formula:

$$\overline{\Delta Q_j'} = \frac{Q_i' + 2Q_m' + Q_f'}{4}.$$

It is highly recommended that the measurement Q'_m be made within the above time constraints so that the first equation can be used in the calculation.

Next, compute the percentage difference in the two average flow rates by

$$d_{1j} = \frac{\overline{\Delta Q'_j} - \overline{\Delta Q_j}}{\overline{\Delta Q'_j}} \times 100 .$$

Report d_{11} , d_{12} , ---, d_{17} and the auditing level on the form in Figure 13 of C below.

4. Calibration Check

Procedure for Performing Check - A calibration check can be made by the same individual and on the same day as the flow-rate check in 3 above. Directions for performing the check are given in D of Section 2.6.

Treatment of Data - Report the percent difference values as determined by the operator (see Section 2.6) in the order that the checks were made as d_{21} , d_{22} , d_{23} , ---, d_{27} , and the auditing level on the form in Figure 13 of C below.

5. Weighing Exposed Filters

Perform the check as instructed in B of Section 2.6 of the Operations Manual. These checks should be made immediately prior to or after the regular weighings. An auditing level of $n=7$ is recommended for lot sizes of $N=50$ to $N=100$, and a level of $n=4$ for lot sizes of $N < 50$. In order that corrections can be made to the lot, it is suggested that lot be made up of filters that are to be weighed at one sitting regardless of how small the number.

In all cases the lot is accepted as good if 1) all check weights are within ± 2.7 mg of the original weights or 2) all filters have been reweighed and corrected.

Report the standard (e.g., ± 2.7 mg) used to judge the weighing process and the auditing level on the form in Figure 13 of C below.

6. Elapsed Time Between Sample Collection and Analysis

Procedure for Performing the Check - For sites where this audit is applicable, the same auditing level and schedule that was set up for flow-rate checks and calibration checks can be used. Directions for performing the check are given in E of Section 2.6 in the Operations Manual.

Treatment of Data - Obtain the delays ($D_1, D_2, \dots, D_7$) in days as reported by the operator and compute

$$d_{3j} = -[0.008 (\% \text{ of OM})(D_j)]^*$$

where

j is the j^{th} check performed during the sampling period,
 $\%$ of OM is the percent organic content of organic matter as given in Table 1 of the Operations Manual, and
 D_j is the delay (days) between collection and analysis for the j^{th} audit check.

Report values of $d_{31}, d_{32}, \dots, d_{37}$ and the auditing level on the form in Figure 13 of C below.

7. Data Processing Check

Perform an independent data processing check on the same samples as were selected for reweighing in 5 above. Directions for performing the check are given in F of Section 2.6 in the Operations Manual.

The lot is accepted without change if all check calculations are within ± 3 percent of the original calculations. If one check calculation differs by more than ± 3 percent from the original, all samples are recalculated and the check calculation reported as the correct concentration of suspended particulates.

Report the standard (e.g., ± 3 percent) used to judge the data processing operation and the auditing level on the form in Figure 13 in C below.

* Derivation of this equation is discussed in Section 3.3.1.

C. Treatment of Collected Information

1. Identification of Defects

One procedure for identifying defects is to evaluate auditing checks in sets, i.e., $d_{11}d_{21}d_{31}$ counts as one set, $d_{12}d_{22}d_{32}$ another, etc., --- $d_{17}d_{27}d_{37}$. If one or more members of any set are defective, it counts as one defect. No more than one defect can be declared per set. Corrigible errors should be corrected when found and are not, therefore, discussed here.

Any set of auditing checks in which the value of d_{1j} , d_{2j} , or d_{3j} is greater than ± 9 will be considered a defect. This value is assumed to be approximately the 3σ value for each of the three parameters. As field data from the auditing program become available, this limit or standard should be reevaluated and adjusted, if necessary. All values of d_{3j} are negative and d_{1j} will be negative most of the time although small positive values may occur occasionally. Values of d_{2j} are expected to be normally distributed with a mean of zero.

2. Reporting Data Quality

Each lot of data submitted with SAROAD forms or tapes should be accompanied by the minimum data qualifying information as shown in Figure 13. The individual responsible for the quality assurance program should sign and date the form. As an illustration, values from Section 3.2, Suggested Standards for Judging Performance, are used to fill in the blanks in Figure 13. The reported auditing rate is the rate in effect at the beginning of the auditing period. An increase or decrease in auditing rate during the auditing period will be reflected by the total number of checks reported. The reason for change should be noted on the form.

Check values (i.e., d_{1j} 's, d_{2j} 's and d_{3j} 's) are calculated as directed in Section 3.1.B and reported as a percent to the nearest whole number. All reported check values exceeding the definition of a defect should be marked for easy recognition by circling on the form.

Attach the data qualification form to the SAROAD form and forward for additional internal review or to the user.

Supervisor's Signature _____ Reporting Date _____

Parameter	Standard Used	Audit Level
Filter Surface Alkalinity	$6.5 \leq \text{pH} \leq 7.5$	$n=7, N=100$
Weighing of Clean Filters	$\pm 1 \text{ mg}$	$n=7, N=100$
Weighing of Exposed Filters	$\pm 2.7 \text{ mg}$	$n=7, N \geq 50$; or $(n=4, N < 50)$
Data Processing Check	$\pm 3\% \text{ of S.P.}^*$	$n=7, N \geq 50$; or $(n=4, N < 50)$
Parameter	Definition of Defect	Audit Level
Flow-Rate Check	$ d_{1j} > 9$	$n=7, N=100$; or $(n=3, N=15)$
Calibration Check	$ d_{2j} > 9$	$n=7, N=100$; or $(n=3, N=15)$
Elapsed Time Between Collection and Analysis	$ d_{3j} > 9$	$n=7, N=100$; or $(n=3, N=15)$

* S.P. = concentration of suspended particulates in $\mu\text{g}/\text{m}^3$ as computed by the operator.

Number of Defects Reported _____ (should be circled in the table below)

Audit	Check Values (percent)						
Flow-Rate Check	d_{11}	d_{12}	d_{13}		d_{1j}		d_{1n}
Calibration Check	d_{21}	d_{22}	d_{23}	-----	d_{2j}	-----	d_{2n}
Elapsed Time Between Collection and Analysis	d_{31}	d_{32}	d_{33}		d_{3j}		d_{3n}

Figure 13: Data Qualification Form

3.1.2 Assessment by Auditing with a Mobile Sampler

An alternate method of auditing the High Volume Method, which in certain situations might be feasible, is to use a mobile sampler as a reference.

A network operating several samplers in a reasonably small area (e.g., city or county) might find this method more convenient than auditing individual variables. However, the reliability of this procedure is directly dependent on the quality of the mobile sampler and how well it is maintained.

For this method a high volume sampler equipped with a continuous flow-rate recorder, a constant voltage regulator, elapsed time indicator, and a constant flow regulator would be maintained by the office of the director and used as a reference. The reference sampler should be operated in accordance with the procedures given in the Operations Manual. For example, Checks 2, 4, 5, 6, and 7 as listed in A of Section 3.1.1 should be made each time the mobile sampler is used. Check 1, filter surface alkalinity, should be audited at least at a level of $n=7$, $N=100$; and only filters from lots where all 7 check values were between 6.5 and 7.5 used. A record should be maintained of the checks performed on the reference sampler and reported with the data if requested by the manager. An audit would be to place the reference sampler adjacent to (but no closer than 3 feet) the field sampler (see Ref. 1 for discussion on positioning the sampler) and sample simultaneously.

The percent difference in the concentration of suspended particulates as measured by the field sampler, $S.P._F$, and the reference sampler, $S.P._R$, is computed by

$$\text{percent difference} = d_j = \frac{S.P._{Fj} - S.P._{Rj}}{0.5(S.P._{Fj} + S.P._{Rj})} \times 100.$$

Based on the results of a collaborative test (Ref. 1) showing a repeatability of the method of 3.0 percent of the mean value, a defect would be defined at the $3\sigma^*$ level as

$$|d_j| \geq 13.$$

* If $\sigma = 3.0$ percent of $S.P.$ for each sampler, then d_j would have a standard deviation of 4.2 percent of the mean value. This gives a 3σ value of approximately 13 percent.

The auditing level for field samplers would be the same as that given in the previous section, i.e., $n=7$, $N=100$.

Only values of d_j 's and the auditing level would need be reported.

3.2 Suggested Standards for Judging Performance Using Audit Data

3.2.1 Suggested Performance Standards for Variables

Suggested standards for judging performance are given in Table 3. Most of these standards are best estimates based on experience and information available in the literature. They should be reevaluated and adjusted as data from the quality assurance program become available. Characteristics of the parameters and variables given in Table 3 are discussed in Section 3.3.

Standards for operation are based on the estimated 1σ , 2σ , and 3σ values for each of the parameters. At the recommended auditing level, i.e., $n=7$, $N=100$, there would be a total of 21 audits in an auditing period. If a normal error distribution is assumed, then only 0.3 percent of the audits would exceed ± 9 or 3σ , 5 percent would exceed ± 6 or 2σ , and only 36 percent would exceed ± 3 or 1σ for a properly operating process. A defect is defined at the 3σ level and should not occur more than once per lot. From the total 21 audits two or more values exceeding the 2σ value (± 6) or 8 or more values exceeding the 1σ value (± 3) for an auditing period indicate a larger-than-normal variance in the data, and correctional changes in the operation should be made.

3.2.2 Suggested Standards for Comparing with Mobile Sampler

Suggested Standards for Defining Defects

1. A value of $|d_j| \geq 13$.

Standard for Audit Levels

2. Suggested minimum auditing rates are: number of audits, $n=7$; lot size, $N=100$; allowable number of defects (i.e., $|d_j| \geq 13$) per lot, $d=0$.

Table 3: Suggested Performance Standards

Parameter	Definition for Defining Defects	Suggested Minimum Standards for Audit Rates
1. Flow-Rate Check	$ d_{1j} > 9$	$n=7, N=100$; or $(n=3, N=15)$
2. Calibration Check	$ d_{2j} > 9$	$n=7, N=100$; or $(n=3, N=15)$
3. Elapsed Time Between Collection and Analysis	$ d_{3j} > 9$	$n=7, N=100$; or $(n=3, N=15)$
Parameter	Standards for Corrigible Errors	Suggested Minimum Standards for Audit Rates
4. Filter Surface Alkalinity	$6.5 \leq \text{pH} \leq 7.5$	$n=7, N=100$
5. Weighing of Clean Filters	$\pm 1 \text{ mg}$	$n=7, N=100$
6. Weighing of Exposed Filters	$\pm 2.7 \text{ mg}$	$n=7, N \geq 50$; or $(n=4, N < 50)$
7. Data Processing Check	$\pm 3\% \text{ of S.P.}$	$n=7, N \geq 50$; or $(n=4, N < 50)$
<u>Standards for Operation</u>		
8. If at any time d^*1 is observed (i.e., a defect is observed for either d_{1j} , d_{2j} , or d_{3j}) increase the audit rate to $n=20, N=100$ or $n=6, N=15$ until the cause has been determined and corrected.		
9. If at any time $d=2$ is observed (i.e., two defects are observed in the same auditing period), cease operation until the cause has been determined and corrected. When data collection resumes, use an auditing level of $n=20, N=100$ (or $n=6, N=15$) until no values greater than ± 6 are observed in three successive audits.		
10. If at any time two (2) values of d_{1j} , d_{2j} , or d_{3j} exceeding ± 6 or three values exceeding ± 3 are observed, 1) increase the audit rate to $n=20, N=100$ or $n=6, N=15$ for the remainder of the auditing period, 2) perform special checks to identify the trouble area, and 3) take necessary corrective action to reduce error levels.		

* d without a subscript as used here represents the number of defects observed in a lot of data.

Suggested Standards for Operation

3. If at any time $|d_j| \geq 13$ increase the auditing rate to $n=20$, $N=100$ until the cause has been determined and corrected.
4. If at any time two (2) values of d_j are observed to exceed 13 during an auditing period, cease gathering data until the cause is determined and corrected. Use an auditing level of $n=20$, $N=100$ when the sampling is resumed until three successive audits are below 8.4.
5. If at any time two (2) values of d_j exceed 8.4 or three (3) values exceed 4.2 in an auditing period, 1) increase the auditing rate to $n=20$, $N=100$, 2) perform special checks to locate trouble areas, and 3) carry out corrective actions to reduce the error level.

3.3 Collection of Information to Detect and Identify Trouble

In a quality assurance program one of the most effective means of preventing trouble is to respond immediately to reports from the operator of suspicious data or equipment malfunctions. Application of proper corrective actions at this point can reduce or prevent the collection of poor quality data. Important error sources, methods for monitoring applicable variables, and suggested control limits for each source are discussed in this section.

3.3.1 Identification of Important Variables

Measurement of the mass of suspended particulate matter in the ambient atmosphere by the High Volume Method requires a sequence of operations and events that yield as an end result a number that serves to represent the average mass of suspended particulates per unit volume of air over the sampling period. Techniques for dynamic calibration of high volume samplers using test atmospheres containing known concentration of particulates are not available. Therefore, there is no way of knowing the accuracy of the values derived from high volume sampling. However, numerous experiments and studies have been performed to identify and evaluate factors which influence the final results. Major sources of

error as identified by a functional analysis of the High Volume Method are discussed below. The parameters are grouped according to whether they influence particulate weight, flow rate, sampling time, or the measured concentration directly. Data processing errors are also discussed.

A. Factors Affecting Particulate Weight

Filter Surface Alkalinity. Flash fired glass-fiber filters are the most frequently used filters for collecting suspended particulate matter for gravimetric analysis. It has been shown (Refs. 2-4) that solid matter is deposited on the fiber surfaces by oxidation of acid gases in the sample air. It was also observed that the quantity of such matter deposited in a given sampling period was not the same for all commercially available glass-fiber filters. Although other reactions are conceivable, the formation of sulfate was studied. It occurs during the first 4 to 6 hours of sampling, and very little is formed after 6 hours (Ref. 2).

Tests conducted with 6.5-pH filters and 11-pH filters showed a significantly larger sulfate to total particulates ratio for the 11-pH filters (Ref. 3). Additional tests (Ref. 4) have shown that alkaline filter media can yield erroneously high results for total particulate matter, sulfates, nitrates, and other species existing as acid gases in the sample air. From samplers operating side by side, one equipped with a pH-11 filter and the other with a pH-6.5 filter, showed after 9 sampling periods that the average total particulate matter was higher by 18 percent, sulfates by 40 percent, and nitrates by 60 percent for pH-11 filters.

The quantity of solid matter deposited during a sampling period is a function of filter pH, length of sampling period or volume of air sampled, and the concentration of acid gases in the sample air. However, even background levels of NO_2 and SO_2 , well below national air quality standards, can induce significant errors when alkaline filters are used.

Relative Humidity Effect. Collected particulates are hygroscopic in varying degrees. Samples collected from suburban, urban, and industrial atmospheres were weighed after being conditioned for a minimum of 4 days at relative humidities varying from 0 to 100 percent (Ref. 5). The results show less than a 1 percent increase in particulate weight in going from 0 to 55 percent relative humidity. However, the relationship is exponential for relative humidities greater than 55 percent, showing a 5 percent increase in particulate weight at a relative humidity of 70 percent and approximately 15 percent weight increase at 80 percent relative humidity. The industrial sample proved most hygroscopic with a 90 percent weight increase at a relative humidity of 100 percent.

The above results point out the importance of maintaining the conditioning environment at a relative humidity less than 55 percent. Also, the humidity level should be the same for conditioning the exposed filter as was used to condition the clean filter. In instances where the exposed filter has to be removed from the conditioning environment for weighing, the time interval between removal and weighing should be kept to a minimum. An interval of less than 5 minutes is recommended.

Elapsed Time Between Sample Collection and Analysis. During the time between sample collection and final weighing volatile matter having substantial vapor pressures may evaporate resulting in a significant reduction in particulate weight.

Results from one set of tests (Ref. 6) indicates that the weight loss is approximately proportional to the percent of organic matter initially present in the collected sample. The greatest rate of loss is experienced during the first 24 hours after collection. A lower but somewhat constant rate of loss continues for several days, the number of which is again a function of the initial content of organic matter.

The equation given for d_{3j} in B of Section 3.1.1 allows one to estimate the possible loss of particulate weight as a function of original organic content and time delay between collection and analysis. This relationship was developed from the data in Reference 6. It shows an approximate loss of 1 percent for a 12-day delay and an initial organic

content of 10 percent, and a weight loss of approximately 6 percent for a 12-day delay of a sample containing 60 percent organic matter.

It is suggested that this could be an important source of error for monitoring sites where the sample is mailed in for analysis and the average organic content of the particulates is greater than 10 percent.

Weighing Errors. Two weighing processes are involved in the High Volume Method. They are the weighing of clean filters and the weighing of exposed filters. If not properly monitored, the weighing process can be a source of significant error in the final result derived from the High Volume Method. Fifty tare-weight weighings for each of five filters made over a period of time in which the relative humidity of the conditioning chamber was varied from 20 to 50 percent showed a maximum variation in tare-weight weighings of 1.2 mg (Ref. 5). Another test showed a standard deviation of approximately 0.8 mg for weighing clean filters after successive 24-hour conditioning periods (Ref. 1). This same test showed a standard deviation of 1.7 mg for weighing exposed filters after successive 24-hour conditioning periods.

These data point out the importance of performing the weighings at the appropriate time, i.e., just after the 24-hour conditioning period, and the necessity of performing the audit or check within a few minutes either before or after the regular weighing in order to expect good agreement between the two weighings.

It is suggested that if the weighing and auditing procedures are properly carried out, the variation between the original and check weights of clean filters should not exceed ± 1.0 mg and not more than ± 2.7 mg for exposed filters.

B. Factors Influencing Flow Rate

Flow-Rate Reading Error. The general feeling of people reading the rotameter is that they can read it to within ± 0.03 m³/min (± 1 ft³/min).

At an average flow rate of 1.13 m³/min (40 ft³/min), this would be equivalent to ± 2.5 percent. Under field conditions this error if not monitored would probably be much greater than 2.5 percent.

Calculating Average Flow Rate. Calculating the average flow rate from initial and final values assuming a constant rate of change throughout the sampling period can result in large errors. One report (Ref. 7) shows an average bias ranging from -1.2 to 8.1 percent of the true average flow rate, for 3 sets of data with 6 samples each. Bias is defined as the difference in average flow rate as computed from the initial and final measurements compared to the average derived from several measurements made throughout the sampling period. These errors can result from particulates plugging the filter resulting in a nonuniform decrease in the flow rate over the sampling period or from variations in source voltage. Nonuniform changes in flow rate are probably greatest in industrial areas due to sticky particulates and can result in a -2 to +10 percent error range in average flow-rate values.

A sampler equipped with a continuous flow-rate recorder does not have the above problem. The true average flow rate can be estimated or calculated by hourly values to within $0.03 \text{ m}^3/\text{min}$ ($1 \text{ ft}^3/\text{min}$) from the recorder chart. This represents a significant improvement in system accuracy.

Flow-Rate Calibration. Calibration of 12 new orifice units by well-qualified individuals using positive displacement meters as primary standards under laboratory conditions showed a standard deviation from the mean of 2.1 percent (Ref. 1). Less qualified people using the orifice unit to calibrate samplers in the laboratory and in the field would be expected to yield a much larger standard deviation. Previous experience with high volume samplers indicate that ± 3 percent of the mean is a reasonable value to use as a standard deviation for calibration error for a well monitored operation. There is also a possible degradation in the calibration with time. Once sufficient field data are available an estimate of its magnitude and characteristics can be made allowing for an optimum calibration schedule to be derived.

Temperature and Pressure Effects on Flow Rate. For most regions in the United States and for a specific elevation, temperatures usually range from -4°C (25°F) to 38°C (100°F) and barometric pressure variations are on the order of $\pm 12.7 \text{ mmHg}$ (0.5 in. Hg) (Ref. 8). Tests on a sampler equipped

with a flow-rate recorder showed a maximum deviation from the calibration curve of +7 percent to -10 percent in the indicated flow rate when going from the extremes of 100°F and 29.0 inches of mercury to 25°F and 30.0 inches of mercury. Calibration conditions were 70°F and 29.5 inches of mercury.

The above data point out the need for either calibrating on site or making corrections for temperature and pressure if the ambient site conditions are significantly different from the laboratory conditions.

C. Sampling Time

Timing Errors. The results of high volume sampling are not very sensitive to the normal magnitudes of timing errors. For example, a 14-minute error in a 24-hr sampling period results in a 1 percent error in the measured concentration. The reference method specifies that times be determined to the nearest 2 minutes. This can be accomplished with the operators' watch or by using an elapsed time indicator on the sampler. In the first instance there is no way of knowing of or compensating for power failures or other interruptions occurring during the sampling period. Samplers equipped with an elapsed time indicator or a continuous flow-rate recorder would indicate such power interruptions and allow one to make corrections.

D. Factors Affecting Measured Concentration Directly

Flow-Rate and Concentration as Functions of Time. - In certain instances when both flow rate and particulate concentration vary during the sampling period, significant errors in the measured average concentration can occur. The example given in Figure 14 is taken as an extreme condition where the concentration of suspended particulates varies from $353 \mu\text{g}/\text{m}^3$ ($10 \mu\text{g}/\text{ft}^3$) to $70.6 \mu\text{g}/\text{m}^3$ ($2 \mu\text{g}/\text{ft}^3$) according to the following equation

$$\text{S.P.} = 141.2 \left(\frac{3}{2} + \cos \frac{\pi}{24} t \right)$$

where

S.P. is the instantaneous concentration in $\mu\text{g}/\text{m}^3$, and
t is the time in hours.

Also, the flow rate decreases from $1.7 \text{ m}^3/\text{min}$ ($60 \text{ ft}^3/\text{min}$) to $1.02 \text{ m}^3/\text{min}$ ($36 \text{ ft}^3/\text{min}$) in a linear fashion according to the following relationship

$$Q = 1.7 - (0.03 \text{ m}^3/\text{hr})t$$

where

Q is the flow rate in m^3/min , and
 t is the time in hours.

The true average concentration, $\overline{\text{S.P.}}$, is seen to be the value at the point where the concentration curve crosses 12 on the time axis, or

$$\overline{\text{S.P.}} = \frac{\int_{t_1}^{t_2} \text{S.P.} dt}{t_2 - t_1} = \frac{\int_0^{24} 141.2 \left(\frac{3}{2} + \cos \frac{\pi}{24} t \right) dt}{24} = 212 \text{ } \mu\text{g}/\text{m}^3 \text{ (6 } \mu\text{g}/\text{ft}^3).$$

However, since the flow rate also varies with time the average concentration as would be measured by the high volume sampler, assuming no other errors are involved, is expressed as:

$$\begin{aligned} \overline{\text{S.P.}}' &= \frac{\int_{t_1}^{t_2} \text{S.P.} Q dt}{\int_{t_1}^{t_2} Q dt} = \frac{\int_0^{24} 141.2 \left(\frac{3}{2} + \cos \frac{\pi}{24} t \right) t (1.7 - 0.03 t) dt}{\int_0^{24} (1.7 - 0.03 t) dt} \\ &= 227 \text{ } \mu\text{g}/\text{m}^3 \text{ (6.42 } \mu\text{g}/\text{ft}^3). \end{aligned}$$

This value differs from the true average concentration by + 7 percent. The reverse case in which the concentration increases as the flow rate decreases is illustrated by the dashed curve in Figure 14 and results in a -6.7 percent deviation from the true average.

In a situation such as that shown in Figure 15 in which the concentration exhibits a diurnal pattern which is symmetrical about the midpoint of the sampling period, the true average concentration is realized by the High Volume Method as long as the flow rate is a linear function of time. In this case

$$S.P. = 353 \left[2 + \sin \frac{\pi}{12} t + \frac{3}{2} \right]$$

and

$$Q = 1.7 - (0.03 \text{ m}^3/\text{hr})t .$$

Performing the same calculations as those done in the example in Figure 14 shows that the "measured" value is the same as the true value.

A deviation greater than ± 7 percent from the true average concentration due to this effect alone should be very rare. The only means of reducing the magnitude of this error is to equip the sampler with a constant flow-rate regulator (Ref. 9). At this time, however, existing constant flow regulators are relatively expensive and are not considered reliable for everyday use in the field.

An estimate of the possible error for a given site could be made by using the local diurnal pattern of suspended particulate concentration and normal or average drop in the flow rate over a 24-hour sampling period to perform the above calculations. The error would not be significant unless the change in flow rate is greater than 20 percent of the initial flow rate, the diurnal pattern is extremely nonsymmetrical about the midpoint of the sampling period, and the maximum concentration is at least four times as great as the minimum.

E. Data Processing Errors

Data processing errors include errors in recording measured values and calculations and in transcribing the calculated values to the SAROAD form. The frequency and magnitude of these errors depend to a great extent on the training and experience of the person performing the task.

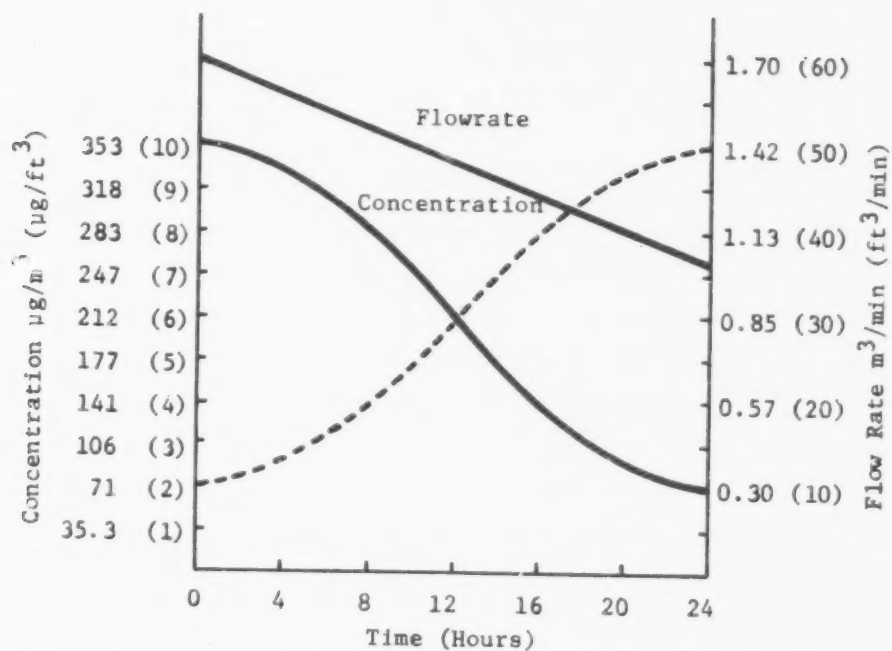


Figure 14: Particulate Concentration and Flow Rate as Functions of Time

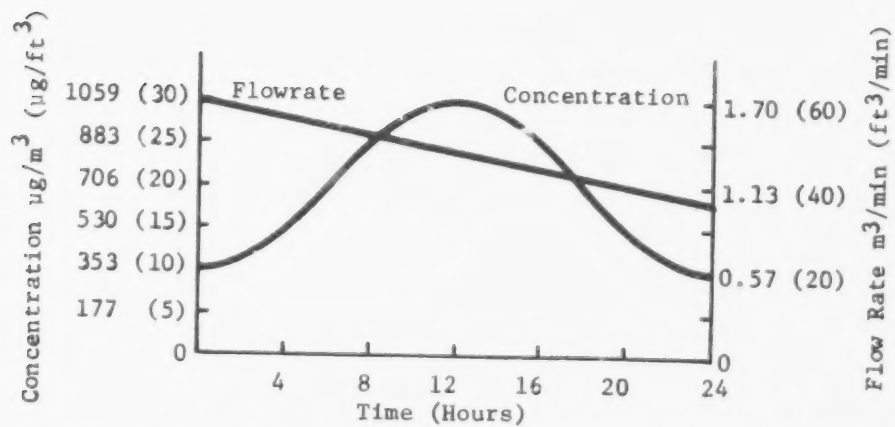


Figure 15: Symmetrical Diurnal Concentration Pattern

An auditing program properly executed should greatly reduce the probability of data processing errors larger than ± 3 percent of the measured concentration getting through the system.

3.3.2 How to Monitor Important Variables

Table 4 summarizes the important variables and how they are or can be monitored. As can be seen from the table, variables 1, 2, 3, 4, and 6 are effectively monitored as part of the suggested auditing program. The relative humidity of the conditioning environment is monitored with a relative humidity indicator or an indicating desiccant as part of the routine operating procedures. Voltage variation would probably be detected as a nonlinear flow-rate drop by the auditing program and could be further monitored with a voltmeter as a special check.

3.3.3 Suggested Control Limits

Appropriate control limits for individual variables will depend on the level of performance needed. Table 5 gives suggested performance standards for determining the average flow rate, calibration error, and loss of particulates due to evaporation of organic matter. The standards as given are no more than estimates of what can be achieved in the field. They should be reevaluated and adjusted as audit data become available.

Suggested control limits for corrigible errors are given in Table 3, Suggested Performance Standards, and are not repeated here.

Combining the means and standard deviations of the three parameters gives a system bias of

$$\text{bias} = \hat{\tau} = \bar{d}_1 + \bar{d}_2 + \bar{d}_3 = -0.06 \times \text{S.P.}$$

and a standard deviation of

$$\hat{\sigma}_T = \sqrt{\sigma_1^2 + \sigma_2^2 + \sigma_3^2} = 0.04 \times \text{S.P.}$$

Table 4: Methods of Monitoring Variables

Variable	Method of Monitoring
1. Filter Surface Alkalinity	Analysis of filters as part of the auditing program.
2. Weighing Process	Reweighing of filters (clean and exposed) as part of the auditing program.
3. Flow Rate Reading Errors	Independent initial and final flow-rate readings performed as part of the auditing program are compared to the operator's regular readings.
4. Nonlinear Flow-Rate Change	Use of the three flow-rate readings made as part of the auditing process to compute an average flow rate and compare to the value derived from two readings.
5. Relative Humidity of the Conditioning Environment	Monitored daily as part of routine operation by use of a relative humidity indicator.
6. Evaporation of Volatile Organic Matter	Monitoring the delay between collection and analysis as part of the auditing process.
7. Voltage Variation	A.C. voltmeter measuring voltage to the sampler and read periodically throughout the sampling period.
8. Data Processing Error	Monitored as part of the auditing process.

It should be noted here that the biases (i.e., $\bar{d}_1$, $\bar{d}_2$, and $\bar{d}_3$) and standard deviations (i.e., S_1 , S_2 , and S_3) as computed in Section 4.1 of the Management Manual are percentages. To arrive at a value in $\mu\text{g}/\text{m}^3$, multiply the decimal equivalent by the S.P. of interest. For example, for an S.P. = $100 \mu\text{g}/\text{m}^3$, the biases given in Table 5 would be

$$\bar{d}_1 = \bar{d}_3 = -0.03 \times 100 = -3 \mu\text{g}/\text{m}^3.$$

Table 5: Suggested Control Limits for Parameters and Variables

Parameter/Variable	Control Limits		
	Mean	Standard Deviation	Upper Limit
1. Flow Rate Check	$\bar{d}_1 = -0.03 \times \text{S.P.}$	$\sigma_1 = 0.02 \times \text{S.P.}$	$\pm 0.09 \text{ S.P.}$
2. Calibration Check	$\bar{d}_2 = 0$	$\sigma_2 = 0.03 \times \text{S.P.}$	$\pm 0.09 \text{ S.P.}$
3. Elapsed Time Between Collection and Analysis (loss or organic material)	$\bar{d}_3 = -0.03 \times \text{S.P.}$	$\sigma_3 = 0.02 \times \text{S.P.}$	$\pm 0.09 \text{ S.P.}$

An overall estimate of data quality would have to include error terms for the corrigible errors as well as the above values. For more details see Part III of this document.

3.4 Procedures for Improving Data Quality

Quality control procedures designed to control or adjust data quality may involve a change in equipment or in operating procedures. Table 6 lists some possible procedures for improving data quality. The applicability or necessity of a procedure for a given monitoring situation will have to be determined from results of the auditing process or special checks performed to identify the important variables. The expected results are given for each procedure in qualitative terms. If quantitative data are available or reasonably good estimates can be made of the expected change in data quality resulting from implementation of each procedure, a graph similar to that in Figure 21, Section 4.3 of the Management Manual can be constructed. The values used in Table 14 and Figure 21 are assumed and were not derived from actual data.

For making cost estimates, a reference system consisting of a sampler equipped with a rotameter and the routine performance of those control checks spelled out in the Operations Manual is assumed.

Equipment, manpower requirements, and the continuing cost of labor and supplies are estimated for each procedure. For these estimates technician time was valued at \$5 per hour and engineering time at \$10 per hour. Equipment life was taken as 5 years. All calculations were based on a sample lot of 100 and an average sampling rate of 60 samples per year per sampling site.

Table 6: Quality Control Procedures or Actions

Procedure/Action	Description of Action	Expected Results	Costs		
			Equip	Personnel	Total
A0. Reference Condition	System using routine procedures as given in the Operations Manual	$\bar{x} = 0.06 \times \text{S.P.}$, $\hat{\sigma}_T = 0.04 \times \text{S.P.}$	---	---	---
A1. Use Continuous Flow-Rate Recorder	Replace the rotameter with a pressure transducer and flow-rate recorder.	Reduce; $\hat{d}_1 = 0$, $\sigma_1 = 0.01 \times \text{S.P.}$ Giving; $\hat{x} = -0.03 \times \text{S.P.}$, $\hat{\sigma}_T = 0.035 \times \text{S.P.}$	\$ 33	None	\$ 33
A2. Install a Constant Voltage Regulator	Install a constant voltage regulator in the power line.	Reduce; $\hat{d}_1 = -0.02 \times \text{S.P.}$, $\sigma_1 = 0.01 \times \text{S.P.}$ Giving; $\hat{x} = -0.05 \times \text{S.P.}$, $\hat{\sigma}_T = 0.035 \times \text{S.P.}$	\$ 90	None	\$ 90
A3. Take 3rd Flow-Rate Reading	Measure, Q_3 , as part of routine operation.	Reduce; $\hat{d}_1 = -0.01 \times \text{S.P.}$, $\sigma_1 = 0.01 \times \text{S.P.}$ Giving; $\hat{x} = -0.04 \times \text{S.P.}$, $\hat{\sigma}_T = 0.035 \times \text{S.P.}$	None	\$ 35	\$ 35
A4. Use Special Mailing for Samples	Use special mailing such as air mail for samples	Reduce; $\hat{d}_3 = -0.01 \times \text{S.P.}$, $\sigma_3 = 0.01 \times \text{S.P.}$ Giving; $\hat{x} = -0.04 \times \text{S.P.}$, $\hat{\sigma}_T = 0.035 \times \text{S.P.}$	None	None	\$ 25
A5. Use Local Laboratory	Use a local laboratory (e.g., college or high school) to condition and weigh samples (implemented as temporary measure only).	Reduce; $\hat{d}_3 = 0$, $\sigma_3 = 0.01 \times \text{S.P.}$ Giving; $\hat{x} = -0.03 \times \text{S.P.}$, $\hat{\sigma}_T = 0.035 \times \text{S.P.}$	\$100	\$100	\$200

A procedure for selecting the appropriate quality control procedure to insure a desired level of data quality is given below:

- 1) Specify the desired performance standard, that is, specify the limits within which you want the deviation between the measured and the true concentration to fall a desired percentage of the time. For example, to measure within $\pm 0.12 \times \text{S.P.}$, 95 percent of the time, the following performance standards must be satisfied:

$$|\hat{\tau} \pm 2\hat{\sigma}_T| \leq 0.12 \times \text{S.P.}$$

- 2) Determine the system's present performance level from the auditing process, as described in Section 4.1 of the Management Manual, by setting

$$\hat{\tau} = \bar{d}_1 + \bar{d}_2 + \bar{d}_3$$

and

$$\hat{\sigma}_T = \sqrt{\sigma_1^2 + \sigma_2^2 + \sigma_3^2}.$$

If the relationship of 1) above is satisfied, no control procedures are required.

- 3) If the desired performance standard is not satisfied, identify the major error components.
- 4) Select the quality control procedure(s) which will give the desired improvement in data quality at the lowest cost. Figure 21 in Section 4.3 of the Management Manual illustrates a method for accomplishing this.

The relative position of actions on the graph in Figure 21 will differ for different monitoring networks according to type of equipment being used, available personnel, and local costs. Therefore, each network would need to develop its own graph to aid in selecting the control procedure providing the desired data quality at the lowest cost.

3.5 Procedures for Changing the Auditing Level to Give the Desired Level of Confidence in the Reported Data

The auditing process does not in itself change the quality of the reported data. It does provide a means of assessing the data quality. An increased auditing level increases the confidence in the assessment. It also increases the overall cost of data collection.

Various auditing schemes and levels are discussed in Section 4.2. Numerous parameters must be known or assumed in order to arrive at an optimum auditing level. Therefore, only two decision rules with two levels of auditing each will be discussed here.

For conditions as assumed in C of Section 4.2 of the Management Manual, a study of Figure 20, page 104, gives the following results. These conditions may or may not apply to your operation. They are included here to call attention to a methodology. Local costs must be used for conditions to apply to your operation.

A. Decision Rule - Accept the Lot as Good If No Defects Are Found (i.e., $d = 0$).

- 1) Most Cost Effective Auditing Level - In Figure 20 the two solid lines are applicable to this decision rule, i.e., $d = 0$. The cost curve has a minimum at $n = 7$ or an auditing level of 7 checks out of 100 sampling periods. From the probability curve it is seen that at this auditing level there is a probability of 0.47 of accepting a lot as good when the lot (for $N = 100$) actually has 10 defects. The associated average cost is 240 dollars per lot.
- 2) Auditing Level for Low Probability of Accepting Bad Data - Increasing the auditing level to $n = 20$, using the same curve in Figure 20 as in (1) above, shows a probability of 0.09 of accepting a lot as good when the lot actually has 10 defects. The average cost associated with this level of auditing is approximately 425 dollars per lot.

B. Decision Rule - Accept the Lot as Good If No More Than One (1) Defect is Found (i.e., $d \leq 1$).

- 1) Most Cost Effective Auditing Level - From the two dashed curves in Figure 20 it can be seen that the cost curve has a minimum at $n = 14$. At this level of auditing there is a probability of 0.55 of accepting a lot of data as good when it has 10 defects. The average cost per lot is approximately 340 dollars.
- 2) Auditing Level for Low Probability of Accepting Bad Data - For an auditing level of $n = 20$ the probability of accepting a lot with 10 percent defects is about 0.36 as read from the $d \leq 1$ probability curve. The average cost per lot is approximately 375 dollars.

It must be realized that the shape of a cost curve is determined by the assumed costs of performing the audit and of reporting bad data. These costs must be determined for individual monitoring situations in order to select optimum auditing levels.

3.6 Monitoring Strategies and Cost

Selecting the optimum monitoring strategy in terms of cost and data quality requires a knowledge of the present data quality, major error components, cost of implementing available control procedures, and potential increase in system precision and accuracy.

Section 4.3 illustrates a methodology for comparing strategies to obtain the desired precision of the data. Table 6 of Section 3.4 lists control procedures with estimated costs of implementation and expected results in terms of which error component(s) are affected by the control. The expected results are estimates and were not derived from actual data.

Three system configurations identified as best strategies in Figure 21 of the Management Manual are summarized here from Section 4.3 of the Management Manual.

Again, local costs and expected results derived from field data are required to select optimum strategies by this method.

A. Reference Method (A0)

Description of Method: This refers to a sampler equipped with a rotameter for making flow-rate measurements. Routine operating procedures as given in the Operations Manual are to be followed with special checks performed to identify problem areas when performance standards are not being met. An auditing level of $n=7$, $N=100$ is to be carried out for this strategy. This method or strategy is identified as A0 in Table 14 and Figure 21 in the Management Manual.

Costs: Taken as reference or zero cost.

Data Quality: Data quality can be described by

$$S.P._T = S.P._m - \hat{t} \pm 3\hat{\sigma}_T$$

where

$S.P._T$ = true average concentration of suspended particulates, and

$S.P._m$ = measured average concentration of suspended particulates.

Taking the hypothesized values of the bias and standard deviation from Table 14 and using in the above relationship shows that for a true concentration, $S.P._T$, of $100 \mu\text{g}/\text{m}^3$, the measured value, $S.P._m$, would fall within the following limits

$$94 < S.P._m < 118$$

approximately 99.7 percent of the time.

B. Modified Reference Method (A1)

Description of Method: This strategy is identical to the reference method in A above except that a pressure transducer and a continuous recorder are used to measure and record the sample air flow rate.

Costs: The average cost per 100 samples is estimated at 33 dollars (see Section 3.4).

Data Quality: From Table 6, values of bias and standard deviation are seen to be $\hat{\tau} = 0.03 \times \text{S.P.}$ and $\hat{\sigma}_T = 0.035 \times \text{S.P.}$. The data quality would be described by

$$\text{S.P.}_T = \text{S.P.}_m - 0.03 \times \text{S.P.}_T \pm 3 \times 0.035 \times \text{S.P.}_T .$$

For a true concentration, S.P._T , of $100 \mu\text{g}/\text{m}^3$ the measured value, S.P._m , would fall within the limits

$$92 < \text{S.P.}_m < 114$$

approximately 99.7 percent of the time.

C. Modified Reference Method Plus Action (A1 + A4)

Description of Method: This method is identified as A1 and A4 in Figure 21 of the Management Manual. This method is the same as B above with the addition of Action A4 which would reduce errors due to loss of organic matter by minimizing time between collection and analysis.

Costs: Average cost per lot is estimated at 58 dollars.

Data Quality: From Table 6 the data quality would be described by

$$\text{S.P.}_T = \text{S.P.}_m - 0.02 \times \text{S.P.}_T \pm 3 \times 0.033 \times \text{S.P.}_T .$$

For a true concentration, S.P._T , of $100 \mu\text{g}/\text{m}^3$ the measured value, S.P._m , would fall within the limits

$$92 < \text{S.P.}_m < 112 .$$

Results from these estimated values show that in going from Method A to Method C, the data spread is decreased by about 16 percent and the range is more evenly distributed about the true concentration value.

PART III. MANAGEMENT MANUAL

4.0 GENERAL

The objectives of a data quality assurance program for the High Volume Method of measuring the concentration of suspended particulate matter in air were given in Section 1.0. In this part of the document, procedures will be given to assist the manager in making decisions pertaining to data quality based on the checking and auditing procedures described in Sections 2.0 and 3.0. These procedures can be employed to:

- 1) detect when the data quality is inadequate,
- 2) assess overall data quality,
- 3) determine the extent of independent auditing to be performed,
- 4) relate costs of data quality assurance procedures to a measure of data quality, and to
- 5) select from the options available to the manager the alternative(s) which will enable him to meet the data quality goals by the most cost-effective means.

Objectives 1 and 2 above* are described in Section 4.1. The determination of the extent of auditing is considered in Section 4.2. Finally, Objectives 4 and 5 are discussed in Section 4.3. The cost data are assumed and a methodology provided. When better cost data become available, improvements can be made in the management decisions.

If the current reference system is providing data quality consistent with that required by the user there will be no need to alter the physical system or to increase the auditing level. In fact several detailed procedures could be bypassed if continuing satisfactory data quality is implied by the audit. However, if the data quality is not adequate, e.g., either a large bias and/or imprecision in the reported data, then (1) increased auditing should be employed, (2) the assignable cause is to be determined, and (3) the system deficiency corrected. The correction can take the form of a change in the operating procedure, e.g., take a mid-point flow-rate reading; or it may be a change in equipment such as

the installation of a constant voltage regulator. An increase in the auditing level will increase the confidence in the reported measure of precision/bias and aid in identifying the assignable cause(s) of the large deviations. The level of auditing will be considered in Section 4.2.

4.1 Data Quality Assessment

The audit procedure and the reported results can serve a two-fold purpose. They can be used to (1) screen the data, by lots of say $N = 50$ or 100 , to detect when the data quality may be inadequate and (2) calculate the bias and precision of the audited measurement and hence estimate the bias/precision of the final reported concentration of suspended particulate matter in the ambient air. In order to perform (1), suggested standards are provided for use in comparing the audited results with the reported values and a defect is defined in terms of the standards. This approach requires only the reporting of the number of defects in the n auditing checks. In the second method above, it is required to report the measures of bias/precision in the audits as will be described below. These values are then used in assessing the overall data quality. Approach (1) is suggested as a beginning step even though it will not make maximum use of the data collected in the auditing program. The simplicity of the approach and the definition of a defective will aid in its implementation. After experience has been gained in using the auditing scheme and in reporting and calculating the results, it is recommended that (2) be implemented.

It is important that the audit procedure be independent of previously reported results and be a true check of the system under normal operating procedures. Independence can be achieved by providing a control sample of unknown concentration to the operator and requesting that he measure and report the concentration of the sample, or having another person perform the check. To insure that the check is made under normal operating procedures, it is required that the audit be performed without any special check of the system prior to the audit other than that usually performed each sampling period.

A. Assessment of Individual Measurements

Assume for convenience that an auditing period consists of $N = 100$ days (or sampling periods). Subdivide the auditing period into n equal periods or nearly equal periods. Make one audit during each period and compute the deviations (differences) between the audit values and the stated values (or previously determined values as measured by the operator) as indicated in the Supervision Manual. For example, if seven audits ($n = 7$) are to be performed over 100 sampling periods ($N = 100$), the 100 periods can be subdivided into 7 intervals (6 with 14 periods and 1 with 16 periods). Select one day at random within each interval and perform the suggested audits. The operator should not be aware of when the checks are to be performed.

For sites operating every sixth day, a minimum of three audits per quarter is recommended. Samples from individual sites can be grouped into logical lots, e.g., all sites for which a single operator is responsible, to form data lots of at least 50 samples. This approach insures that the audit level will exceed $n = 7$ for the combined sites and resulting data.

In order to assess the data quality using measures of bias/precision, the checks are to be combined for the selected auditing period and the mean difference or bias and the standard deviation of the differences are to be computed as indicated below.

The formulas for average bias and the estimated standard deviations are the standard ones given in statistical texts (e.g., see Ref. 10). The level of sampling or auditing, n , will be considered as a parameter to be selected by the manager to assess the quality of data as required.

1) Flow-Rate Checks

$$\text{Bias} = \bar{d}_1 = \frac{\sum_{j=1}^n d_{1j}}{n},$$

where

d_{1j} = percentage deviation of average flow rates, $\bar{\Delta Q}$ and $\bar{\Delta Q}'$, as determined from the two-point and three-point approximations (see page 55 of Section 3.1).

$$\text{Standard Deviation} = s_1 = \sqrt{\frac{\sum (d_{1j} - \bar{d}_1)^2}{n-1}},$$

where

$\bar{d}_1$ = the average bias, and

s_1 = the estimated standard deviation of the average flow rate corrected for the average bias $\bar{d}_1$.

2) Calibration Check

$$\text{Bias} = \bar{d}_2 = \frac{\sum_{j=1}^n d_{2j}}{n},$$

where

d_{2j} = deviation of the measured flow rates determined by operator and by one performing the audit,

$$\text{Standard Deviation} = s_2 = \sqrt{\frac{\sum (d_{2j} - \bar{d}_2)^2}{2(n-1)}}^*.$$

3) Elapsed Time Between Sample Collection and Analysis

In order to compute an overall bias and standard deviation associated with vaporization of organic matter when there is a time delay between sample collection and analysis, use the values of d_{3j} as reported by the operator (see 6, page 57) and calculate

$$\text{Bias} = \bar{d}_3 = \frac{\sum_{j=1}^n d_{3j}}{n}, \text{ and}$$

$$\text{Standard Deviation} = s_3 = \sqrt{\frac{\sum (d_{3j} - \bar{d}_3)^2}{n-1}}$$

*The factor 2 is inserted in the denominator to account for the fact that the variance of the difference of two measurements, each with the same variance, is twice the variance of an individual measurement.

Individual checks on the standard deviations of the three audits can be made by computing the ratio of the estimated standard deviation, s_1 , to the corresponding suggested performance standard, σ_1 , given in Table 7. If this ratio exceeds values given in Table 7 for any one of the audits, this would indicate that the source of trouble may be assigned to that particular aspect of the measurement process. Critical values of this ratio are given in Figure 16 as a function of sample size and two levels of confidence. Having assessed the general problem area, one then needs to perform the appropriate quality control checks to determine the specific causes of the large deviations.

Table 7. Critical Values of s_1/σ_1

Level of Confidence	Statistic	Audit Level				
		n=5	n=10	n=15	n=20	n=25
90%	s_1/σ_1	1.40	1.29	1.23	1.20	1.18
95%	s_1/σ_1	1.54	1.37	1.30	1.26	1.23

s_1 = estimated standard deviation

σ_1 = hypothesized or suggested standard deviation

Audit	Suggested Performance Standard
Flow Rate Check	$\sigma_1 = 0.02 \times \text{S.P.}$
Calibration Check	$\sigma_2 = 0.03 \times \text{S.P.}$
Elapsed Time Between Collection and Analysis	$\sigma_3 = 0.02 \times \text{S.P.}$
Overall Standard Deviation	$\sigma_T = 0.041 \times \text{S.P.}$

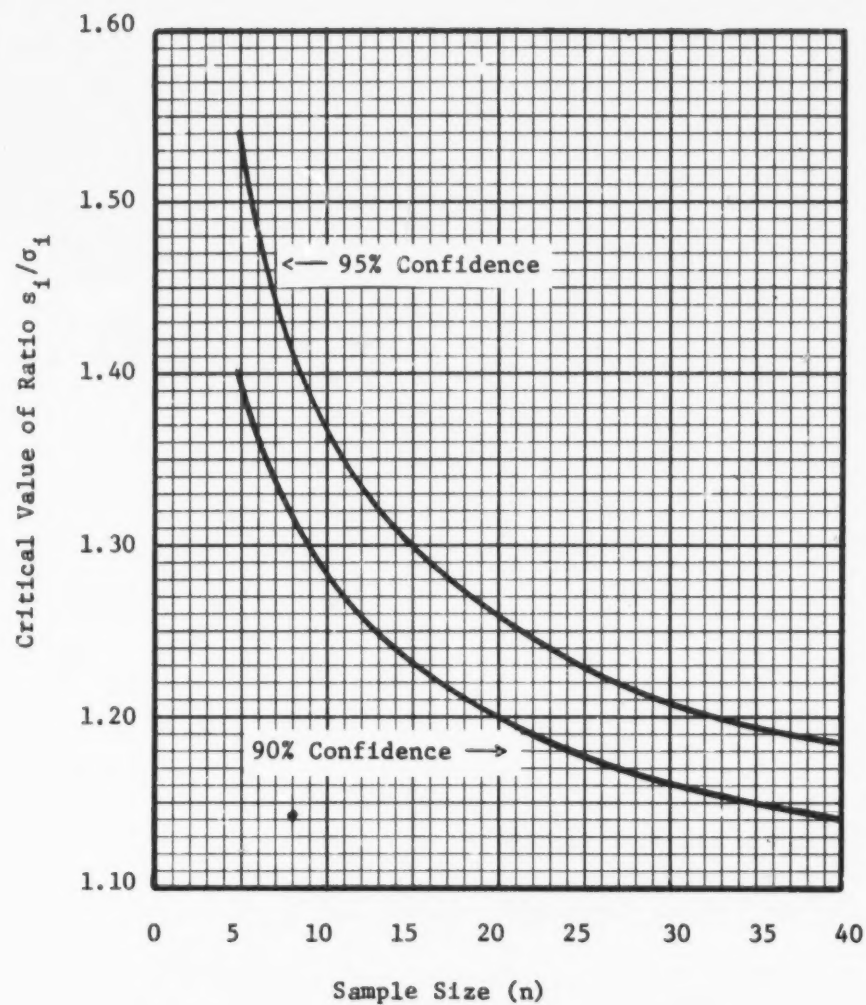


Figure 16: Critical Values of Ratio s_1/σ_1 Vs. n

B. Overall Assessment of Data Quality

The values $\bar{d}_1$, $\bar{d}_2$, and $\bar{d}_3$, s_1 , s_2 , and s_3 above measure the bias and variation of the reported data for the three audits considered. The biases and standard deviations of the remaining variables can be estimated from the suggested standards under the assumption that the data quality is consistent with the standards, or they may be obtained by determining the effects of each bias and standard deviation on the reported concentrations S.P.

1) Development of a Model

In order to be able to make objective decisions concerning the High Volume Method for measuring the concentration of particulate matter in air, it was helpful to develop a mathematical model of the process since there is no way of generating a standard atmosphere to calibrate the sampler. The measurement of particle concentration is dependent on several parameters, operator effects, environmental conditions, calibration procedures, variation in instrumentation, and other variables and effects, some of which are perhaps unknown to us. In developing the model, data were collected from several publications and exploratory experiments. If data were not available, engineering judgement concerning the magnitude of the effects was used. Starting with the basic deterministic equation for estimating the particle concentration ($\mu\text{g}/\text{m}^3$), an effects model was developed to include all of the parameters, variables, and errors which could be identified as possible contributors to the variation in the results. Ten error terms are included in the model. The model and the estimated effects of each of the parameters in the model are discussed in further detail in the Final Report on this contract.

2) Identification of the Important Parameters

The next step in the modeling process was to use the model to identify the critical parameters, i.e., those parameters which may cause the greatest variation in the concentration, S.P., if their variation is

of the order of magnitude assumed in the analysis. Two types of analyses were employed to determine the critical parameters and the combined effect of all of the parameters on the variation in the measured concentration, S.P.

The first type was a sensitivity or ruggedness analysis which identified and ranked the critical parameters, made certain checks on the adequacy of a linear approximation to the developed model, and estimated the variation (as measured by the standard deviation) of S.P. through the use of a linear approximation. This latter technique was a straightforward application of error analysis. The second analysis procedure was a Monte Carlo simulation in which each of the parameters was assigned a distribution of values; for example, the weighing error was assumed to be normally distributed with given mean and standard deviation. This simulation analysis provided a listing of the simulated values of concentration in ascending order and calculated the mean and standard deviation and other pertinent characteristics of this distribution. These analyses are described in some detail in the Final Report of this contract.

Results from the above analyses may not be valid for one specific situation, but should be a reasonably good evaluation of average precision and accuracy obtainable over a large population of samplers. The results indicate that if the operating procedures recommended in the Operations Manual were adhered to, the measured data would have a mean value very close to the true value (i.e., there would be no bias, $\hat{\tau} = 0$) and a standard deviation of approximately 6 percent of the mean value ($\hat{\sigma}_T \approx 0.06 \times \text{S.P.}$). This held true for simulated concentrations ranging from about $50 \mu\text{g}/\text{m}^3$ to $300 \mu\text{g}/\text{m}^3$.

Values derived from the above analyses were used to arrive at suggested performance standards, and to a certain extent, for suggested control limits given for certain checks in the Operations Manual.

The standard deviation of S.P. is a measure of the precision or variation of the reported values of S.P. as estimated by the model. It is to be noted that this measure depends on the estimated standard deviations of each of the variables and on the coefficients in the model, which are dependent on the form of the model. These values can be

checked using the biases and standard deviations computed from actual field data. The true concentration of suspended particulates should fall in the following interval where $S.P._m$ is the measured concentration,

$$S.P._m - \hat{\tau} \pm 2\hat{\sigma}_T^* ,$$

approximately 95 percent of the time, or within the interval

$$S.P._m - \hat{\tau} \pm 3\hat{\sigma}_T ,$$

approximately 99.7 percent of the time. When computed from audit data, the value $2\hat{\sigma}_T$ is actually dependent on the number of audits conducted. If n is large, say about 25 or larger, the value 2 is appropriate.

In reporting the data quality, the bias, overall standard deviation, and auditing level should be reported in an ideal situation (see Section 4.4 for further discussion on data presentation). More restricted information is suggested in the Supervision Manual as a minimal reporting procedure.

If the overall reported percisions/biases of the data meet or satisfy the requirements of the user of the data, then a reduced auditing level may be employed; on the other hand, if the data quality is not adequate, assignable causes of large deviations should be determined and appropriate action taken to correct the deficiencies. This determination may require an increased checking or auditing of the measurement process as well as the performance of certain quality control checks, e.g., monitor voltage variations over 24-hour sampling period.

4.2 Auditing Schemes

Auditing a measurement process costs time and money. On the other hand, reporting poor quality data can also be very costly. For example, the reported data might be used to determine a relationship between health damage and concentrations of certain pollutants. If poor quality

* A positive bias in the measurement must be subtracted from the measured value when estimating the true concentration.

data are reported, it is possible that invalid inferences or standards derived from the data will cost many dollars. These implications may be unknown to the manager until some report is provided to him referencing his data; hence, the importance of reporting the precision and bias with the data.

As a result of the cost of reporting poor quality data it is desirable to perform the necessary audits to assess the data quality and to invalidate unsatisfactory data with high probability. On the other hand, if the data quality is satisfactory, an auditing scheme will only increase the data measurement and processing cost. An appropriate tradeoff or balance of these costs must be sought. These costs are discussed in Section C below.

Now consider the implication of an auditing scheme to determine or judge the quality of the reported data in terms of an acceptance sampling scheme. Let the data be assembled into homogeneous lots of $N = 50$ or 100 sampling periods. Suppose that n periods are sampled in the manner suggested in Section 4.1. That is, the $N = 50$ or 100 sampling periods are subdivided into equal time intervals (as nearly equal as possible) then one day is selected at random during each interval. Figure 17 gives a diagram of the data flow, sampling, and decision making process for an auditing level of $n = 7$.

A. Statistics of Various Auditing Schemes

Suppose that the lot size is $N = 100$ periods (days), that $n = 7$ periods are selected at random, and that there are 5% defectives in the 100, or 5 defectives. The probability that the sample of 7 contains 0, 1, ..., 5 defectives is given by the following.

$$p(0 \text{ defectives}) = \frac{\binom{5}{0} \binom{95}{7}}{\binom{100}{7}}^*$$

and for d defectives

$$p(d \text{ defectives}) = \frac{\binom{5}{d} \binom{95}{7-d}}{\binom{100}{7}}, d \leq 5.$$

The values are tabulated below for $d = 0, 1, \dots, 6$ and for the two data quality levels.

Table 8: P(d defectives)

d	Data Quality	
	D=5% Defectives	D=15% Defectives
0	0.6903	0.3083
1	0.2715	0.4098
2	0.0362	0.2152
3	0.0020	0.0576
5	0.00004	0.0084
6	= 0	≈ 0

*

Figure 18A gives the probabilities of $d = 0$ and $d \leq 1$ defectives as a function of sample size. The probability is given for lot size $N = 100$, $D = 5$ and 15% defectives, for sample sizes (auditing levels) from 1 to 25. For example, if $n = 10$ measurements are audited and $D = 5\%$ defectives, the probability of $d=0$ defectives is 0.58. Figure 18B gives the probabilities for lot size $N = 50$, for $D = 6, 10$, and 20% defectives, and for $d = 0$ and $d \leq 1$. These curves will be used in calculating the cost relationships of Section C.

$$* \frac{\binom{5}{0} \binom{95}{7}}{\binom{100}{7}} = \frac{\left(\frac{5!}{0!5!}\right) \left(\frac{95!}{7!88!}\right)}{\left(\frac{100!}{7!93!}\right)} = \frac{95 \cdot 94 \cdots 89}{100 \cdot 99 \cdots 94} = 0.6903.$$

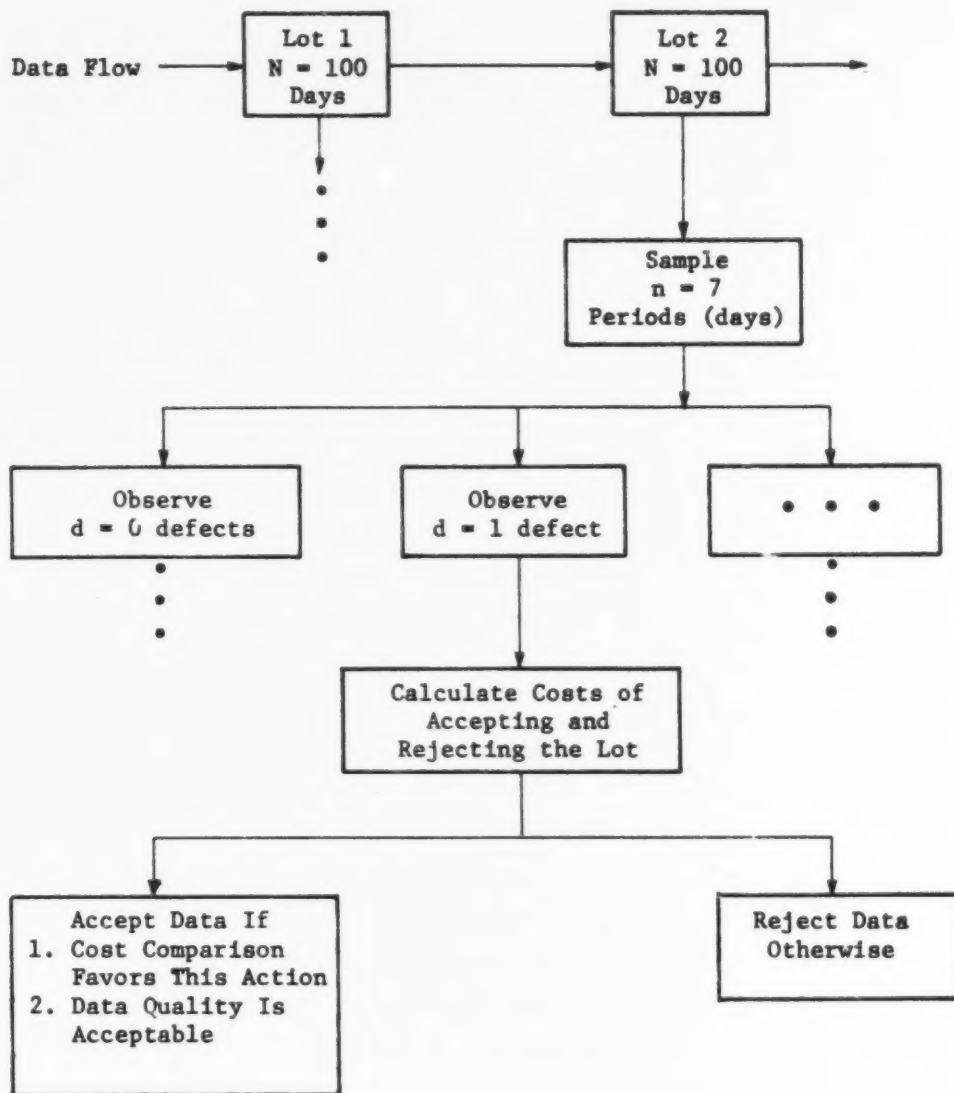


Figure 17: Data Flow Diagram for Auditing Scheme

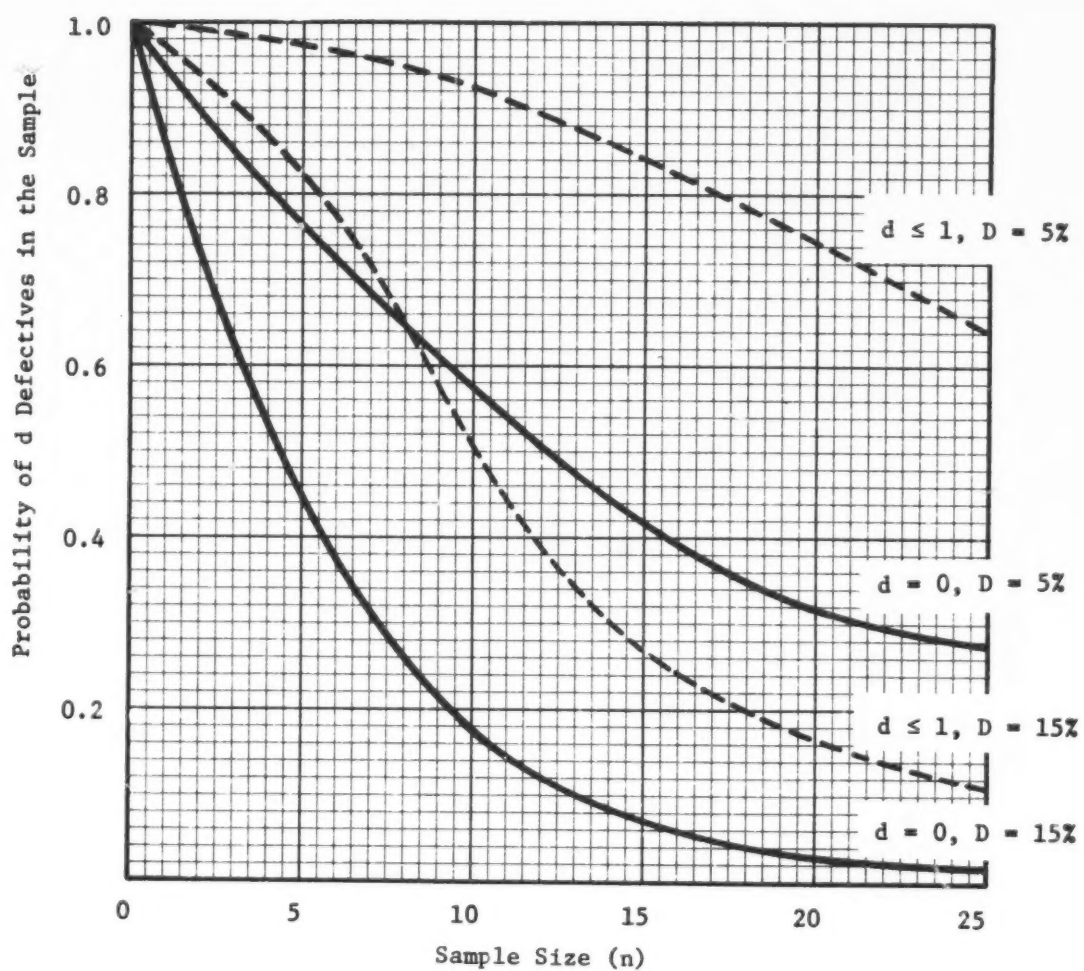


Figure 18A: Probability of d Defectives in the Sample If
the Lot ($N = 100$) Contains $D\%$ Defectives

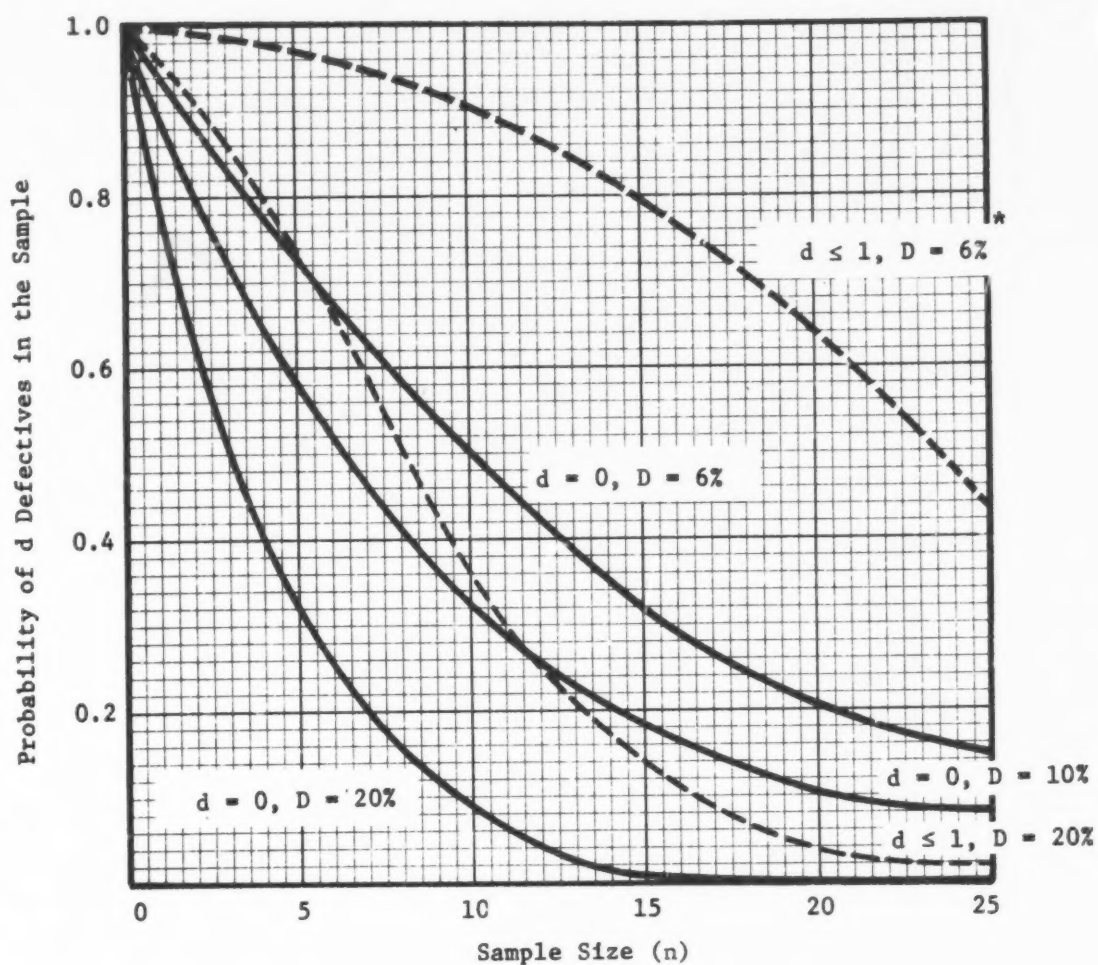


Figure 16B: Probability of d Defectives in the Sample If the
Lot ($N = 50$) Contains $D\%$ Defectives.

* This graph is for a lot size of $N = 50$. Only whole numbers of defectives are physically possible; therefore, even values of D (i.e., 6, 10, and 20 percent) are given rather than the odd values of 5 and 15 percent as given in Figure 18A.

B. Selecting the Auditing Level

One consideration in determining an auditing level n used in assessing the data quality is to calculate the value of n which for a prescribed level of confidence will imply that the percent of defectives in the lot is less than ten percent, say, if zero defectives are observed in the sample.* Figures 19A and 19B give the percentage of good measurements in the lot sampled for several levels of confidence, 50, 60, 80, 90, and 95%. The curves in 19A assume that 0 defectives are observed in the sample, and 19B, 1 defective observed in the sample. The solid curves on the figures are based on a lot size of $N = 100$; two dashed curves are shown in Figure 19A for $N = 50$; the differences between the corresponding curves are small for the range of sample sizes considered.

For example, for zero defectives in a sample of 7 from a lot of $N = 100$, one is 50% confident that there are less than 10% defective measurements among the 100 reported values. For zero defectives in a sample of 15 from $N = 100$, one is 80% confident that there are less than 10% defective measurements. Several such values were obtained from Figure 19A and placed in Table 9 below for convenient reference.

Table 9: Required Auditing Levels n
for Lot Size $N = 100$
Assuming Zero Defectives

Confidence Level	D = 10%	15%	20%
50%	7	<5	<5
60%	9	6	<5
80%	15	10	8
90%	20	15	11
95%	≈ 25	18	13

* Obviously, the definition of defective need not always be the same and must be clearly stated each time. The definitions employed herein are based on results of collaborative test programs.

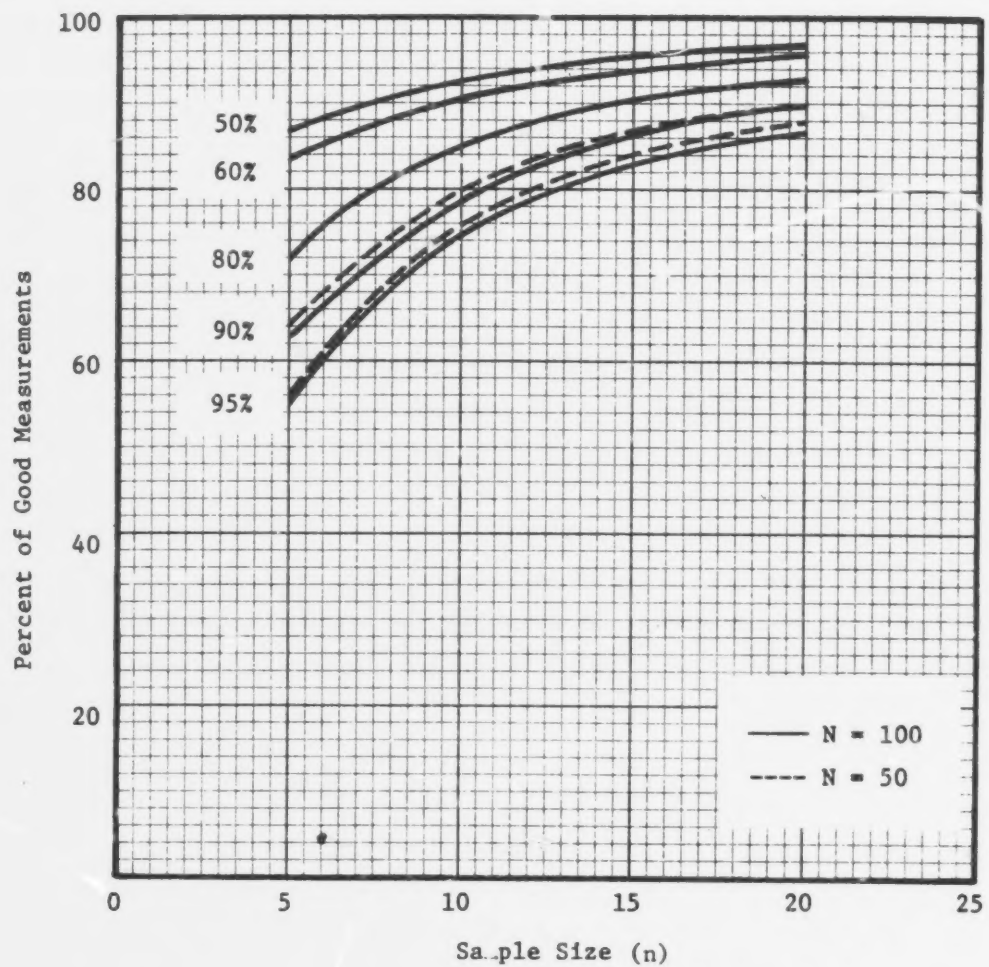


Figure 19A: Percentage of Good Measurements Vs. Sample Size
for No Defectives and Indicated Confidence Level

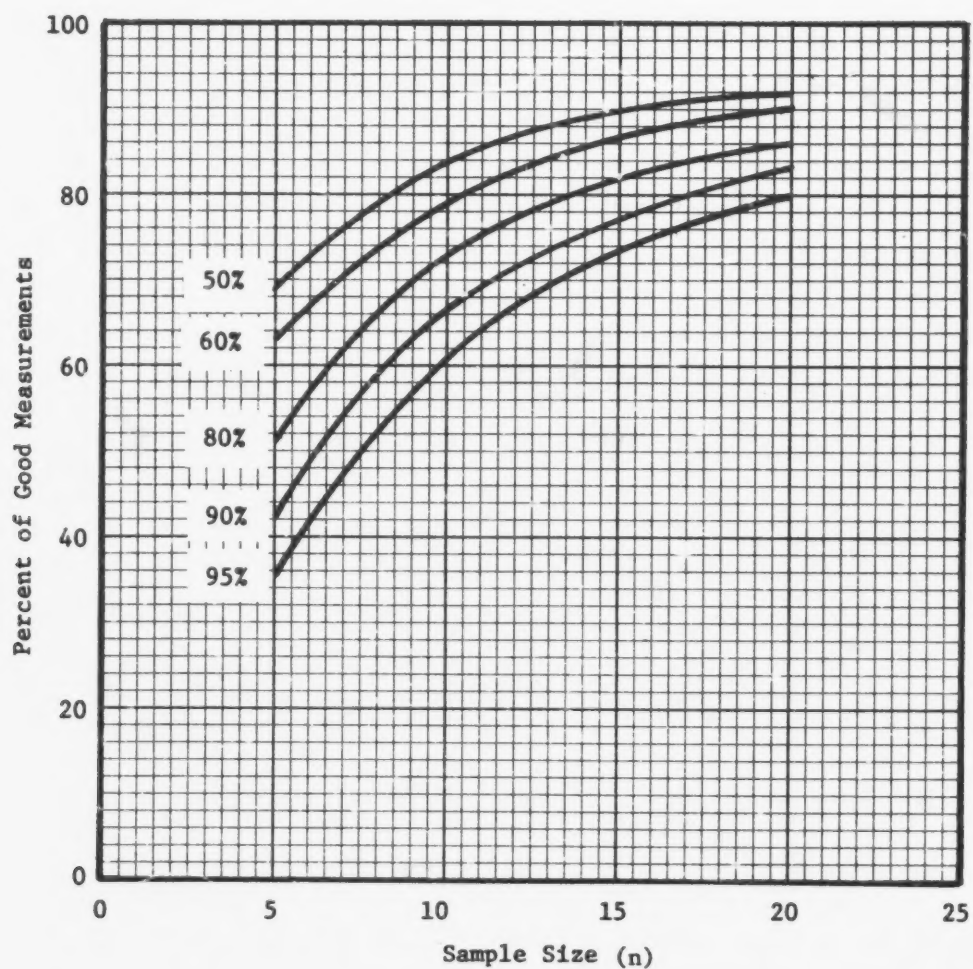


Figure 19B: Percentage of Good Measurements Vs. Sample Size

for 1 Defective Observed and Indicated Confidence Level

Lot Size = 100

C. Cost Relationships

The auditing scheme can be translated into costs using the costs of auditing, rejecting good data, and accepting poor quality data. These costs may be very different in different geographic locations. Therefore, purely for purposes of illustrating a method, the cost of auditing is assumed to be directly proportional to the auditing level. For $n = 7$ it is assumed to be \$155 per lot of 100. The cost of rejecting good quality data is assumed to be \$600 for a lot of $N = 100$. The cost of reporting poor quality data is taken to be \$800. To repeat, these costs given in Table 10 are assumed for the purpose of illustrating a methodology of relating auditing costs to data quality. Meaningful results can only be obtained by using correct local information.

Table 10: Costs vs. Data Quality

	<u>Data Quality</u>	
	<u>"Good"</u> $D \leq 10\%$ <u>Incorrect Decision</u>	<u>"Bad"</u> $D > 10\%$ <u>Correct Decision</u>
Reject Lot of Data	Lose cost of performing* audit plus cost of rejecting good quality data. (-\$600 - \$155)	Lose cost of performing audit, save cost of not permitting poor quality data to be reported. (\$400 - \$155)
Accept Lot of Data	<u>Correct Decision</u> Lose cost of performing audit. (-\$155)	<u>Incorrect Decision</u> Lose cost of performing audit plus cost of declaring poor quality data valid. (-\$800 - \$155)

* Cost of performing audit varies with the sample size; is assumed to be \$155 for $n = 7$ audits per $N = 100$ lot size.

Suppose that 50 percent of the lots have more than 10 percent defective and 50 percent have less than 10 percent defective. (The percentage of defective lots can be varied as will be described in the final report.) For simplicity of calculation, it is further assumed that the good lots have exactly 5 percent defectives and the poor quality lots have 15 percent defective.

Suppose that $n = 7$ measurements out of a lot $N = 100$ have been audited and none found to be defective. Furthermore, consider the two possible decisions of rejecting the lot and accepting the lot and the relative costs of each. These results are given in Tables 11A and 11B.

Table 11A: Costs If 0 Defectives are Observed and the Lot is Rejected

		Correct Decision	Incorrect Decision	Net Value (\$)
Reject Lot	D = 5%	---	$p_1 = 0.69$ $C_1 = -600 - 155$	$p_1 C_1 = -\$521$
	D = 15%	$p_2 = 0.31$ $C_2 = 400 - 155$	---	$p_2 C_2 = \$76$

$$\text{Cost} = p_1 C_1 + p_2 C_2 = -\$445$$

Table 11B: Costs If 0 Defectives are Observed and the Lot is Accepted

		Correct Decision	Incorrect Decision	Net Value (\$)
Accept Lot	D = 5%	$p_1 = 0.69$ $C_3 = -155$	---	$p_1 C_3 = -\$107$
	D = 15%	---	$p_2 = 0.31$ $C_4 = -800 - 155$	$p_2 C_4 = -\$296$

$$\text{Cost} = p_1 C_3 + p_2 C_4 = -\$403$$

The value $p_1(p_2)$ in the above table is the probability that the lot is 5% (15%) defective given that 0 defectives have been observed. For example,

$$p_1 = \frac{\left(\begin{array}{c} \text{probability that the lot is 5\% defective} \\ \text{and 0 defectives are observed} \end{array} \right)}{p \left(\begin{array}{c} \text{lot is 5\% defective and} \\ \text{0 defectives observed} \end{array} \right) + p \left(\begin{array}{c} \text{lot is 15\% defective and} \\ \text{0 defectives observed} \end{array} \right)}$$

$$= \frac{0.5(0.69)}{0.5(0.69) + 0.5(0.31)} = 0.69.$$

$$p_2 = \frac{\left(\begin{array}{c} \text{probability that the lot is 15\% defective} \\ \text{and 0 defectives are observed} \end{array} \right)}{p \left(\begin{array}{c} \text{lot is 5\% defective and} \\ \text{0 defectives observed} \end{array} \right) + p \left(\begin{array}{c} \text{lot is 15\% defective and} \\ \text{0 defectives observed} \end{array} \right)}$$

$$= \frac{0.5(0.31)}{0.5(0.31) + 0.5(0.69)} = 0.31.$$

It was assumed that the probability that the lot is 5% defective is 0.5. The probability of observing zero defectives, given the lot quality is 5% or 15%, can be read from the graphs of Figures 18A or 18B.

A similar table can be constructed for 1, 2, ..., defectives and the net costs determined. The net costs are tabulated in Table 12 for 1, 2, and 3 defectives. The resulting costs indicate that the decision preferred from a purely monetary viewpoint is to accept the lot if 0 defectives are observed and to reject it otherwise. The decision cannot be made on this basis alone. The details of the audit scheme also affect the confidence which can be placed in the data qualification; consideration must be given to that aspect as well as to cost.

Table 12: Costs in Dollars

Decision	d = number of defectives			
	0	1	2	3
Reject Lot	-445	-155	+101	+207
Accept Lot	-403	-635	-839	-928

D. Cost Vs. Audit Level

After the decision criteria have been selected, an average cost can be calculated. Based on the results of Table 12, the decision criterion is to accept the lot if $d = 0$ defectives are observed and to reject the lot if $d = 1$ or more defectives are observed. All the assumptions of the previous section are retained. The auditing level is later varied to obtain the data in Figure 20.

One example calculation is given below and summarized in Table 13. The four cells of Table 13 consider all the possible situations which can occur, i.e., the lots may be bad or good and the decision can be to either accept or reject the lot based on the rule indicated by Table 12. The costs are exactly as indicated in Tables 11A and 11B. The probabilities are computed as follows.

$$\begin{aligned} q_1 &= (\text{prob. that the lot is 5\% defective and 1 or} \\ &\quad \text{more defects are obtained in the sample}) \\ &= (\text{prob. that the lot is 5\% defective})(\text{prob. 1 or} \\ &\quad \text{more defectives are obtained in the sample} \\ &\quad \text{given the lot is 5\% defective}) \\ &= 0.5 (0.31) = 0.155 \end{aligned}$$

Similarly q_2 , q_3 , and q_4 in Table 13 are obtained as indicated below.

$$q_2 = 0.5 (0.69) = 0.345$$

$$q_3 = 0.5 (0.69) = 0.345$$

$$q_4 = 0.5 (0.31) = 0.155$$

The sum of all the q 's must be unity as all possibilities are considered. The value 0.5 in each equation is the assumed proportion of good lots (or poor quality lots). The values 0.31 and 0.69 are the conditional probabilities that given the quality of the lot, either $d = 0$ or $d = 1$ or more defectives are observed in the sample. Further details of the computation are given in the final report of this contract.

Table 13: Overall Average Costs for One
Acceptance - Rejection Scheme

Decision	Good Lots D = 5%	Bad Lots D = 15%	
Reject any lot of data if 1 or more defects are found.	$q_1 = 0.155$ $C_1 = -\$755$	$q_2 = 0.345$ $C_2 = \$245$	$q_1 C_1 + q_2 C_2 = -\$32$
Accept any lot of data if 0 defects are found.	$q_3 = 0.345$ $C_3 = -\$155$	$q_4 = 0.155$ $C_4 = -\$955$	$q_3 C_3 + q_4 C_4 = -\$202$

Average Cost = $-\$234$

In order to interpret the concept of average cost, consider a large number of data lots coming through the system; a decision will be made on each lot in accordance with the above and a resulting cost of the decision will be determined. For a given lot, the cost may be any one of the four costs, and the proportion of lots with each cost is given by the q 's. Hence the overall average cost is given by the sum of the product of q 's by the corresponding C 's.

In order that one may relate the average cost as given in Table 13 to the costs given in Table 12, it is necessary to weight the costs in Table 12 by the relative frequency of occurrence of each observed number of defectives, i.e., $\text{prob}(d)$. This calculation is made below.

No. of Defectives	Decision Rule	Costs (\$) from Table 12	Prob(d)	Cost $\times$ Prob(d)
d = 0	Accept	- 403	0.50	$-\$201.5$
1	Reject	- 155	0.34	- 52.7
2	Reject	101	0.1255	12.6
3	Reject	207	0.030	6.2
4	Reject	244	0.0042	1.0
	Totals		0.9997	$-\$234.4$

Thus the value -\$234 is the average cost of Table 13 and the weighted average of the costs of Table 12. The weights, $\text{Prob}(d)$, are obtained as follows:

$$\begin{aligned}\text{Prob}(d=0) &= \text{Prob}(\text{lot is good and } d=0 \text{ defectives are observed}) \\ &+ \text{Prob}(\text{lot is poor quality and } d=0 \text{ defectives are observed}) \\ &= 0.5(0.69) + 0.5(0.31) = 0.50.\end{aligned}$$

This is the proportion of all lots which will have exactly 0 defectives under the assumptions stated. For $d = 1, 2, 3$, and 4, the values of the probabilities in parentheses above can be read from Table 8.

Based on the stated assumptions the average cost was determined for several auditing levels as indicated in Table 13. These costs are given in Figure 20. One observes from this figure that $n = 7$ is cost effective given that one accepts only if zero defectives are observed. (See curve for $d = 0$.)

If the lots are accepted if either 0 or 1 defectives are observed, then referring to the curve $d \leq 1$, the best sampling level is $n = 15$. The curve of probability of $d = 0$ ($d \leq 1$) defectives in a lot of $N = 100$ measurements if there are 10% defectives, is also given on the same figure.

Another alternative is to accept all data without performing an audit. Assuming that one-half (50%) of the lots contain more than 10% defectives, the average cost on a per lot basis would be $0.5(-\$800) = -\$400$. This, however, would preclude qualification of the data. Regardless of cost, it would be an unacceptable alternative.

4.3 Data Quality Versus Cost of Implementing Actions

The discussion and methodology given in the previous section were concerned with the auditing scheme (i.e., level of audit or sample size, costs associated with the data quality, etc.). Increasing the level of audit of the measurement process does not by itself change the quality of the data, but it does increase the information about the

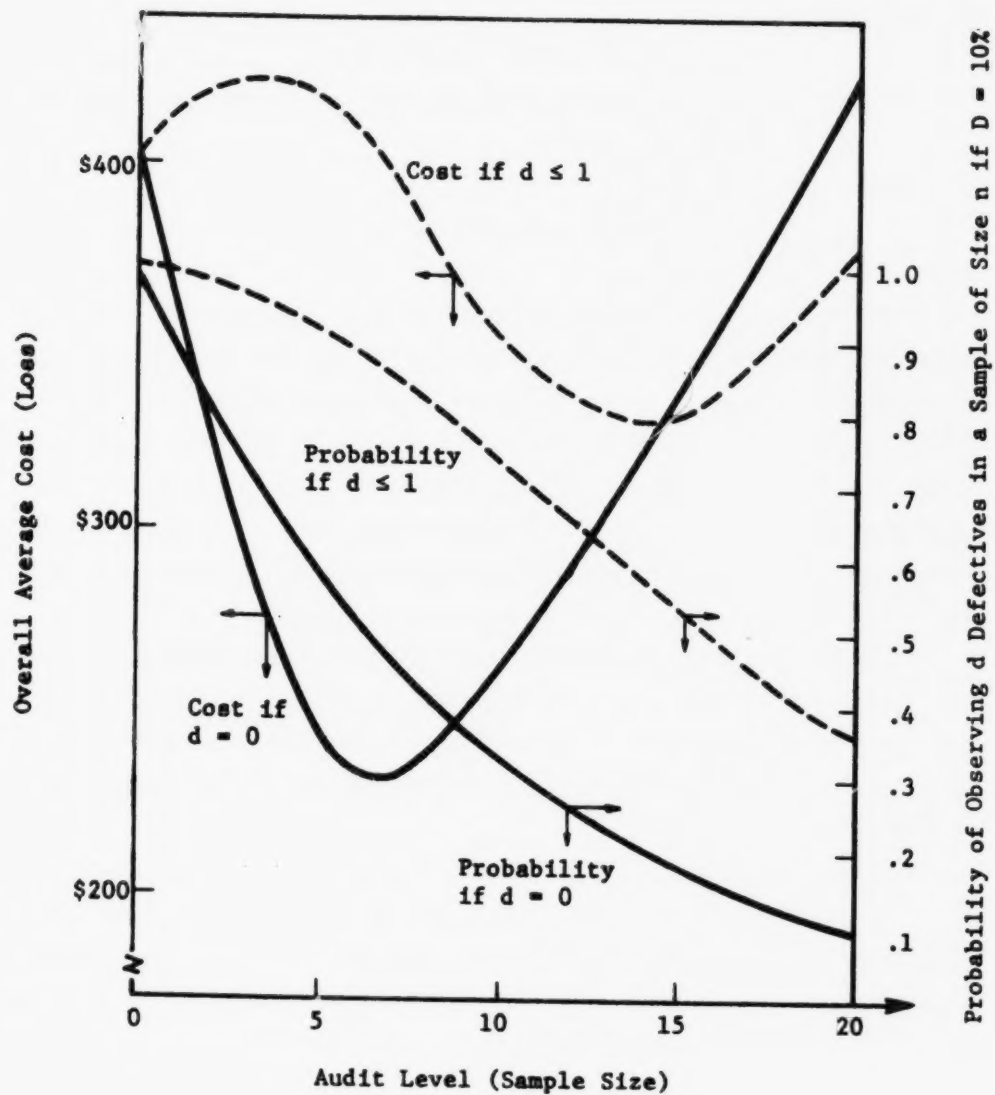


Figure 20: Average Cost Vs. Audit Level
(Lot Size $N = 100$)

quality of the reported data. Hence, fewer good lots will be rejected and more poor quality data will be rejected. If the results of the audit imply that certain process measurement variables are major contributors to the total error or variation in the reported S.P., then alternative strategies for reducing these variations need to be investigated. This section illustrates a methodology for comparing the strategies to obtain the desired precision of the data. In practice it would be necessary to experiment with one or more strategies, determine the potential increase in precision, relate the precisions to the relative costs as indicated herein. Several strategies are considered, but only a few of the least costly ones would be acceptable as illustrated in Figure 21. The assumed values of the standard deviations and biases for each type audit are not based on actual data, except for the reference method. In this case values were taken from Ref. 1. These values are probably smaller than those experienced in the field.

Several alternative actions or strategies can be taken to increase the precision of the reported data. For example, if the voltage variations are large, the flow rate will vary and, depending upon the diurnal variation, will cause variation in S.P. Similarly the nature of the particulate matter may cause a large decrease in the flow rate. Under these conditions additional control equipment for one or more of the environmental effects can reduce the variation of the measured responses by calculated amounts and thus reduce the error of the reported concentrations. In this manner, the cost of the added controls can be related to the data quality as measured by the estimated bias/precision of the reported results. Because there is a significant bias, the measure of variation of the reported results is taken as the square root of the mean square error, i.e., $M = \sqrt{\sigma_T^2 + \tau^2}$.

In order to determine a cost efficient procedure, it is necessary to estimate the variance for each source of error (or variation) for each strategy and then select the strategy or combination of strategies which yield the desired precision with minimum cost. These calculations are summarized in Table 14 with assumed costs of equipment and control procedures.

Suppose that it is desired to make a statement that the true S.P. is within $12 \mu\text{g}/\text{m}^3$ with approximately 95 percent confidence. Minimal cost control equipment and checking procedures are to be employed to attain this desired precision.

Examining the graph in Figure 21 of cost versus precision, one observes that A4 is the least costly strategy that meets the required goal of $2M = 0.12$ or $M = 0.06$ (i.e., an overall error of 6% of S.P.) in the reported concentration. Similarly the combination of A1 and A4 meets the requirement that $3M = 0.12$ or $M = 0.04$ (i.e., an overall error of 4% of S.P.). The assumed values of the standard deviations of the measured concentrations of suspended particulates for the alternative courses of action are given in Table 14. The costs for the various alternatives are given in Table 6 of Section 3.4 and in Table 14.

Suppose that it is desired that M be less than 0.04 and that the cost of reporting poor quality data increases rapidly for M greater than 0.04. This assumption is illustrated by the cost curve given by the solid line in Figure 21. For any alternative strategy, the cost of reporting poor quality data is given by the ordinate of this curve corresponding to the strategy.

Table 14: Assumed Standard Deviations and Biases for Alternative Strategies

		Alternative Strategies						
		A0	A1	A2	A3	A4	A5	A1+A4
1. Flow Rate Check	$\bar{d}_1$	0.03*	0	0.03	0.01	0.03	0.03	0
	σ_1	0.02	0.01	0.01	0.01	0.02	0.02	0.01
2. Calibration Check	$\bar{d}_2$	0	0	0	0	0	0	0
	σ_2	0.03	0.03	0.03	0.03	0.03	0.03	0.03
3. Elapsed Time Between Sample Collection and Analysis	$\bar{d}_3$	0.03	0.03	0.03	0.03	0.01	0	0.01
	σ_3	0.02	0.02	0.02	0.02	0.01	0.01	0.01

** σ_T	0.041	0.037	0.037	0.037	0.037	0.037	0.033
*** Bias = τ	0.06	0.04	0.05	0.04	0.04	0.03	0.01
$M = \sqrt{\sigma_T^2 + \tau^2}$	0.073	0.048	0.062	0.054	0.054	0.048	0.035
Added Cost (\$)/100 Samples	0	33	90	35	25	200	58

Alternative Strategies are given in Table 6, Section 3.4, the σ_i 's, $i = 1, 2$, and 3 , are assumed values based on results given in Ref. 1, and where data are not available, they are engineering judgments.

* All of these values are percent error, i.e., 0.03 is equivalent to $0.03 \times \text{S.P.}$, etc. for each value given.

** $\sigma_T^2 = \sigma_1^2 + \sigma_2^2 + \sigma_3^2$, $\sigma_T = \sqrt{\sigma_T^2}$.

*** Bias = $\tau = \bar{d}_1 + \bar{d}_2 + \bar{d}_3$.

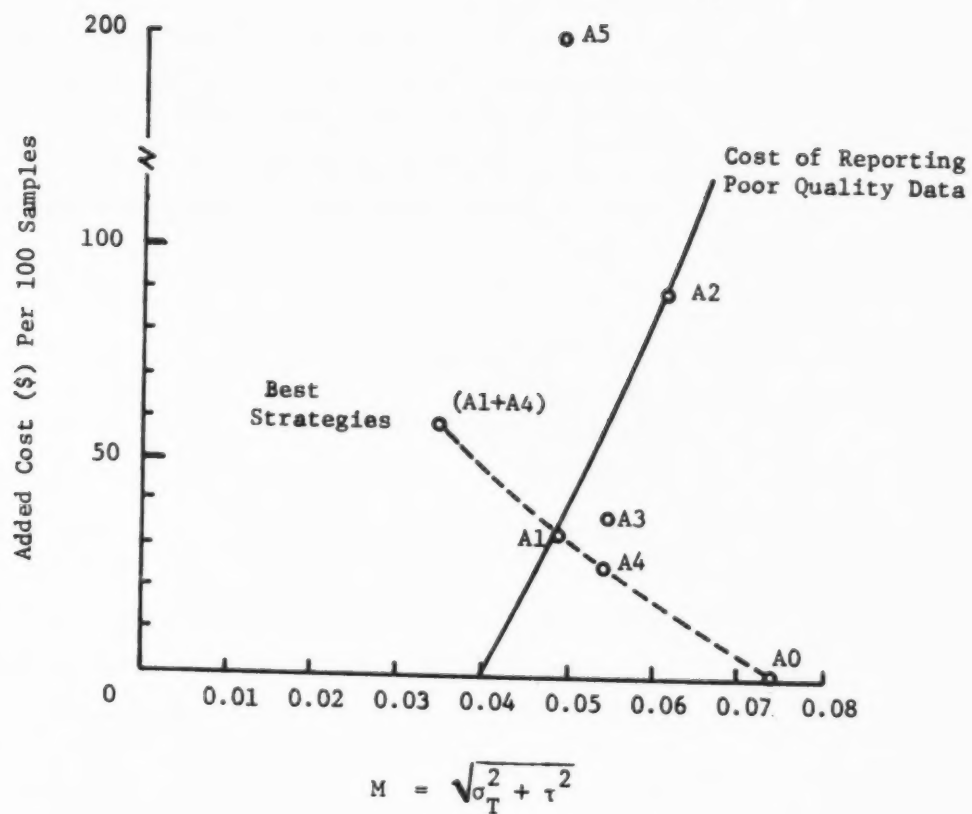


Figure 21: Added Costs Vs. $\sqrt{\sigma_T^2 + \tau^2}$ for Alternative Strategies

4.4 Data Presentation

A reported value whose precision and accuracy (bias) are unknown is of little, if any, worth. The actual error of a reported value--that is, the magnitude and sign of its deviation from the true value--is usually unknown. Limits to this error, however, can usually be inferred, with some risk of being incorrect, from the precision of the measurement process by which the reported value was obtained and from reasonable limits to the possible bias of the measurement process. The bias, or systematic error, of a measurement process is the magnitude and direction of its tendency to measure something other than what was intended; its precision refers to the closeness or dispersion of successive independent measurements generated by repeated applications of the process under specified conditions, and its accuracy is determined by the closeness to the true value characteristic of such measurements.

Precision and accuracy are inherent characteristics of the measurement process employed and not of the particular end result obtained. From experience with a particular measurement process and knowledge of its sensitivity to uncontrolled factors, one can often place reasonable bounds on its likely systematic error (bias). This has been done in the model for the measured concentration as indicated in Table 14. It is also necessary to know how well the particular value in hand is likely to agree with other values that the same measurement process might have provided in this instance or might yield on measurements of the same magnitude on another occasion. Such information is provided by the estimated standard deviation of the reported value, which measures (or is an index of) the characteristic disagreement of repeated determinations of the same quantity by the same method and thus serves to indicate the precision (strictly, the imprecision) of the reported value.

A reported result should be qualified by a quasi-absolute type of statement that places bounds on its systematic error and a separate statement of its standard deviation, or of an upper bound thereto, whenever a reliable determination of such value is available. Otherwise a computed value of the standard deviation should be given together with a statement of the number of degrees of freedom on which it is based.

As an example, consider strategy A0 in Table 14 of Section 4.3. Here, the assumed standard deviation and bias are; $\sigma_T = 0.041 \times \text{S.P.}$ and $\tau = 0.06 \times \text{S.P.}_T$, respectively, where S.P._T is the true concentration of suspended particulates. The results would be reported as the measured concentration, S.P._m , with the following 2σ limits and audit level, e.g.,

$$\text{S.P.}_m - 0.06 \times \text{S.P.} \pm 0.082 \times \text{S.P.}; n=7, N=100.$$

4.5 Personnel Requirements

Personnel requirements as described here are in terms of the High Volume Method only. It is realized that these requirements may be only a minor factor in the overall requirements from a systems point-of-view where several measurement methods are of concern simultaneously.

A. Training and Experience

1. Director

The director or one of the professional level employees should have a basic understanding of statistics as used in quality control. He should be able to perform calculations, such as the mean and standard deviation, required to define data quality. The importance of and requirements for performing independent and random checks as part of the auditing process must be understood. Three references which treat the above mentioned topics are listed below:

Probability and Statistics for Engineers, Irvin Miller and John E. Freund, published by Prentice-Hall, Inc., Englewood, N. J., 1965.

Introductory Engineering Statistics, Irwin Guttman and S. S. Wilks, published by John Wiley and Sons, Inc., New York, N. Y., 1965.

The Analysis of Management Decisions, William T. Morris, published by Richard D. Irwin, Inc., Homewood, Illinois, 1964.

2. Operator

The High Volume Method is simple at the operational level requiring no high level skills. A high school graduate with proper supervision and on-the-job training can become a fully capable operator within one month or less.

An effective on-the-job training program could be as follows:

- a) Observe experienced operator perform the different tasks in the measurement process.
- b) Study the operational manual of this document and use it as a guide for performing the operations.
- c) Perform operations under the direct supervision of an experienced operator.
- d) Perform operations independently but with a high level of quality control checks utilizing the technique described in the section on operator proficiency evaluation procedures to encourage high quality work.

Another alternative would be to have the operator attend an appropriate basic training course sponsored by EPA.

4.6 Operator Proficiency Evaluation Procedures

One technique which may be useful for early training and qualification of operators is a system of rating the operators as indicated below.

Various types of violations (e.g., invalid sample resulting from operator carelessness, failure to maintain records, use of improper equipment, or calculation error) would be assigned a number of demerits depending upon the relative consequences of the violation. These demerits could then be summed over a fixed period of time of one week, month, etc. and a continuous record maintained. The mean and standard deviation of the number of demerits per week, can be determined for each operator and a quality control chart provided for maintaining a record of proficiency of each operator and whether any changes in this level have occurred. In comparing operators, it is necessary to assign demerits on a per unit work load basis in order that the inferences drawn from the chart be consistent. *It is not necessary or desirable for the operator to be aware of this form of evaluation. The supervisor should use it as a means of determining when and what kind of instructions and/or training is needed.*

A sample QC chart is given in Figure 22 below. This chart assumes that the mean and standard deviation of the number of demerits per week, e.g., are 5 and 1 respectively. After several operators have been evaluated for a few weeks, the limits can be checked to determine if they are both reasonable and effective in helping to improve or maintain data quality.

The limits should be based on the operators whose proficiency is average or slightly better than average. Deviations outside the QC limits, either above or below, should be considered in evaluating the operators. Identifying those operators whose proficiency may have improved is just as important as knowing those operators whose proficiency may have decreased.

The above procedure may be extended to an entire monitoring network (system). With appropriate definitions of work load, a continuous record may be maintained of demerits assigned to the system. This procedure might serve as an incentive for teamwork, making suggestions for improved operation procedures, etc.

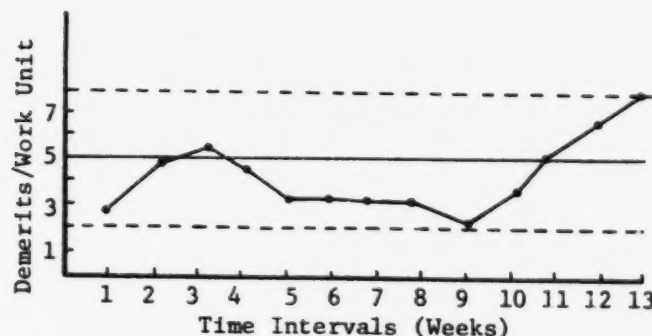


Figure 22: Sample QC Chart for Evaluating Operator Proficiency

REFERENCES

1. Herbert C. McKee et al., "Collaborative Study of Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method)," Southwest Research Institute, Contract CAP 70-40, SwRI Project 21-2811, San Antonio, Texas, June 1971.
2. Robert E. Lee, Jr. and Jack Wagman, "A Sampling Anomaly in the Determination of Atmospheric Sulfate Concentration," American Industrial Hygiene Association Journal 27, pp. 266-271, May-June 1966.
3. Rober M. Burton et al., "Field Evaluation of the High-Volume Particle Fractionating Cascade Impactor--A Technique for Respirable Sampling," presented at the 65th Annual Meeting of the Air Pollution Control Association, June 18-22, 1972.
4. Peter K. Mueller et al., "Selection of Filter Media: An Annotated Outline," presented at the 13th Conference on Methods in Air Pollution and Industrial Hygiene Studies, University of California, Berkeley, California, October 30-31, 1972.
5. G. P. Tierney and W. D. Conner, "Hygroscopic Effects on Weight Determinations of Particulates Collected on Glass-Fiber Filters," American Industrial Hygiene Association Journal 28, pp. 363-365, July-August, 1967.
6. John F. Kowalczyk, "The Effects of Various Pre-Weighing Procedures on the Reported Weights of Air Pollutants Collected by Filtration," presented at the 60th Annual Meeting of the Air Pollution Control Association, Cleveland, Ohio, June 11-16, 1967.
7. C. D. Robson and K. E. Foster, "Evaluation of Air Particulate Sampling Equipment," American Industrial Hygiene Association Journal 23, pp. 404-410, 1962.
8. John S. Henderson, "A Continuous-Flow Recorder for the High-Volume Air Sampler," presented at the 8th Conference on Methods in Air Pollution and Industrial Hygiene Studies, Oakland, California, February 6-8, 1967.
9. Walter K. Harrison et al., "Constant Flow Regulators for the High-Volume Air Sampler," American Industrial Hygiene Association Journal 21, pp. 115-120, 1960.
10. Kendall, M. B., The Advanced Theory of Statistics, Vol. I, p. 148-151, Charles Griffic & Company, Ltd., 1948.

APPENDIX A

REFERENCE METHOD FOR THE DETERMINATION OF
SUSPENDED PARTICULATES IN THE ATMOSPHERE
(HIGH VOLUME METHOD)

Reproduced from Appendix B, "National Primary and Secondary Ambient Air Standards," *Federal Register*, Vol 36, No. 84, Part II, Friday, April 30, 1971.

APPENDIX B—REFERENCE METHOD FOR THE
DETERMINATION OF SUSPENDED PARTICULATES
IN THE ATMOSPHERE (HIGH VOLUME
METHOD)

1. Principle and Applicability.

1.1 Air is drawn into a covered housing and through a filter by means of a high-flow-rate blower at a flow rate (1.13 to 1.70 m.³/min.; 40 to 60 ft.³/min.) that allows suspended particles having diameters of less than 100 μ m. (Stokes equivalent diameter) to pass to the filter surface. (1) Particles within the size range of 100 to 0.1 μ m. diameter are ordinarily collected on glass fiber filters. The mass concentration of suspended particulates in the ambient air (μ g./m.³) is computed by measuring the mass of collected particulates and the volume of air sampled.

1.2 This method is applicable to measurement of the mass concentration of suspended particulates in ambient air. The size of the sample collected is usually adequate for other analyses.

2. Range and Sensitivity.

2.1 When the sampler is operated at an average flow rate of 1.70 m.³/min. (60 ft.³/min.) for 24 hours, an adequate sample will be obtained even in an atmosphere having concentrations of suspended particulates as low as 1 μ g./m.³. If particulate levels are unusually high, a satisfactory sample may be obtained in 6 to 8 hours or less. For determination of average concentrations of suspended particulates in ambient air, a standard sampling period of 24 hours is recommended.

2.2 Weights are determined to the nearest milligram, airflow rates are determined to the nearest 0.05 m.³/min. (1.5 ft.³/min.), times are determined to the nearest 2

minutes, and mass concentrations are reported to the nearest microgram per cubic meter.

3. Interferences.

3.1 Particulate matter that is oily, such as photochemical smog or wood smoke, may block the filter and cause a rapid drop in airflow at a nonuniform rate. Dense fog or high humidity can cause the filter to become too wet and severely reduce the airflow through the filter.

3.2 Glass-fiber filters are comparatively insensitive to changes in relative humidity, but collected particulates can be hygroscopic. (2)

4. Precision, Accuracy, and Stability.

4.1 Based upon collaborative testing, the relative standard deviation (coefficient of variation) for single analyst variation (repeatability of the method) is 8.0 percent. The corresponding value for multilaboratory variation (reproducibility of the method) is 3.7 percent. (3)

4.2 The accuracy with which the sampler measures the true average concentration depends upon the constancy of the airflow rate through the sampler. The airflow rate is affected by the concentration and the nature of the dust in the atmosphere. Under these conditions the error in the measured average concentration may be in excess of ± 50 percent of the true average concentration, depending on the amount of reduction of airflow rate and on the variation of the mass concentration of dust with time during the 24-hour sampling period. (4)

5. Apparatus.

5.1 Sampler.

5.1.1 Sampler. The sampler consists of three units: (1) the faceplate and gasket, (2) the filter adapter assembly, and (3) the motor unit. Figure B1 shows an exploded view of these parts, their relationship to each

other, and how they are assembled. The sampler must be capable of passing environmental air through a 406.5 cm.² (63 in.²) portion of a clean 30.3 by 35.4 cm. (8- by 10-in.) glass-fiber filter at a rate of at least 1.70 m.³/min. (60 ft.³/min.). The motor must be capable of continuous operation for 24-hour periods with input voltages ranging from 110 to 120 volts, 50-60 cycles alternating current and must have third-wire safety ground. The housing for the motor unit may be of any convenient construction so long as the unit remains airtight and leak-free. The life of the sampler motor can be extended by lowering the voltage by about 10 percent with a small "buck or boost" transformer between the sampler and power outlet.

5.1.2 Sampler Shelter. It is important that the sampler be properly installed in a suitable shelter. The shelter is subjected to extremes of temperature, humidity, and all types of air pollutants. For these reasons the materials of the shelter must be chosen carefully. Properly painted exterior plywood or heavy gauge aluminum serve well. The sampler must be mounted vertically in the shelter so that the glass-fiber filter is parallel with the ground. The shelter must be provided with a roof so that the filter is protected from precipitation and debris. The internal arrangement and configuration of a suitable shelter with a gable roof are shown in Figure B2. The clearance area between the main housing and the roof at its closest point should be 500.5 $\pm$ 103.5 cm.² (80 $\pm$ 30 in.²). The main housing should be rectangular, with dimensions of about 29 by 36 cm. (11 $\frac{1}{2}$ by 14 in.).

5.1.3 Rotameter. Marked in arbitrary units, frequently 0 to 70, and capable of being calibrated. Other devices of at least comparable accuracy may be used.

8.1.4 Orifice Calibration Unit. Consisting of a metal tube 7.6 cm. (3 in.) ID and 18.9 cm. (6 1/4 in.) long with a static pressure tap 8.1 cm. (3 in.) from one end. See Figure B3. The tube end nearest the pressure tap is flanged to about 10.8 cm. (4 1/4 in.) OD with a male thread of the same size as the inlet end of the high-volume air sampler. A single metal plate 9.3 cm. (3 1/2 in.) in diameter and 0.34 cm. (1/8 in.) thick with a central orifice 2.9 cm. (1 1/4 in.) in diameter is held in place at the air inlet end with a female threaded ring. The other end of the tube is flanged to hold a loose female threaded coupling, which screws onto the inlet of the sampler. An 18-hole metal plate, an integral part of the unit, is positioned between the orifice and sampler to simulate the resistance of a clean glass-fiber filter. An orifice calibration unit is shown in Figure B3.

8.1.5 Differential Manometer. Capable of measuring to at least 40 cm. (16 in.) of water.

8.1.6 Positive Displacement Meter. Calibrated in cubic meters or cubic feet, to be used as a primary standard.

8.1.7 Barometer. Capable of measuring atmospheric pressure to the nearest mm.

8.2 Analysis

8.2.1 Filter Conditioning Environment. Balance room or desiccator maintained at 15° to 35°C and less than 50 percent relative humidity.

8.2.2 Analytical Balance. Equipped with a weighing chamber designed to handle unfolded 203 by 25.4 cm. (8 by 10-in.) filters and having a sensitivity of 0.1 mg.

8.2.3 Light Source. Frequently a table of the type used to view X-ray films.

8.2.4 Numbering Device. Capable of printing identification numbers on the filters.

6 Reagents

6.1 Filter Media. Glass-fiber filters having a collection efficiency of at least 99 percent for particles of 0.3 µm diameter, as measured by the DOP test, are suitable for the quantitative measurement of concentrations of suspended particulates. (5) although some other medium, such as paper, may be desirable for some analyses. If a more detailed analysis is contemplated, care must be exercised to use filters that contain low background concentrations of the pollutant being investigated. Careful quality control is required to determine background values of these pollutants.

7 Procedure

7.1 Sampling

7.1.1 Filter Preparation. Expose each filter to the light source and inspect for pinholes, particles, or other imperfections. Filters with visible imperfections should not be used. A small brush is useful for removing particles. Equilibrate the filters in the filter conditioning environment for 34 hours. Weigh the filters to the nearest milligram; record tare weight and filter identification number. Do not bend or fold the filter before collection of the sample.

7.1.2 Sample Collection. Open the shelter, loosen the wing nuts, and remove the faceplate from the filter holder. Install a numbered, preweighed, glass-fiber filter in position (rough side up); replace the faceplate without disturbing the filter, and fasten securely. Undertightening will allow air leakage; overtightening will damage the sponge-rubber faceplate gasket. A very light application of talcum powder may be used on the sponge-rubber faceplate gasket to prevent the filter from sticking. During inclement weather the sampler may be removed to a protected area for filter change. Close the roof of the shelter, run the sampler for about 5 minutes, connect the rotameter to the nipple on the back of the sampler, and read the rotameter ball with rotameter in a vertical position. Estimate to the nearest whole number. If the ball is fluctuating rapidly, tip the rotameter and slowly straighten it

until the ball gives a constant reading. Disconnect the rotameter from the nipple; record the initial rotameter reading and the starting time as date on the filter folder. (The rotameter should never be connected to the sampler except when the flow is being measured.) Sample for 34 hours from midnight to midnight and take a final rotameter reading. Record the final rotameter reading and ending time and date on the filter folder. Remove the faceplate as described above and carefully remove the filter from the holder, touching only the outer edges. Fold the filter lengthwise so that only surfaces with collected particulates are in contact, and place in a manila folder. Record on the folder the filter number, location, and any other factors, such as meteorological conditions or reading of nearby buildings, that might affect the results. If the sample is defective, void it at this time. In order to obtain a valid sample, the high-volume sampler must be operated with the same rotameter and tubing that were used during its calibration.

7.2 Analysis. Equilibrate the exposed filters for 34 hours in the filter conditioning environment, then reweigh. After they are weighed, the filters may be saved for detailed chemical analysis.

7.3 Maintenance

7.3.1 Sampler Motor. Replace brushes before they are worn to the point where motor damage can occur.

7.3.2 Faceplate Gasket. Replace when the margins of samples are no longer sharp. The gasket may be sealed to the faceplate with rubber cement or double-sided adhesive tape.

7.3.3 Rotameter. Clean as required, using alcohol.

8 Calibration

8.1 Purpose. Since only a small portion of the total air sampled passes through the rotameter during measurement, the rotameter must be calibrated against actual airflow with the orifice calibration unit. Before the orifice calibration unit can be used to calibrate the rotameter, the orifice calibration unit itself must be calibrated against the positive displacement primary standard.

8.1.1 Orifice Calibration Unit. Attach the orifice calibration unit to the intake end of the positive displacement primary standard and attach a high-volume motor blower unit to the exhaust end of the primary standard. Connect one end of a differential manometer to the differential pressure tap of the orifice calibration unit and leave the other end open to the atmosphere. Operate the high-volume motor blower unit so that a series of different, but constant, airflows (usually six) are obtained for definite time periods. Record the reading on the differential manometer at each airflow. The different constant airflows are obtained by placing a series of loadplates, one at a time, between the calibration unit and the primary standard. Placing the orifice before the inlet reduces the pressure at the inlet of the primary standard below atmospheric; therefore, a correction must be made for the increase in volume caused by this decreased inlet pressure. Attach one end of a second differential manometer to an inlet pressure tap of the primary standard and leave the other open to the atmosphere. During each of the constant airflow measurements made above, measure the true inlet pressure of the primary standard with this second differential manometer. Measure atmospheric pressure and temperature. Correct the measured air volume to true air volume as directed in 9.1.1, then obtain true airflow rate, Q , as directed in 9.1.3. Plot the differential manometer readings of the orifice unit versus Q .

8.1.2 High-Volume Sampler. Assemble a high-volume sampler with a clean filter in place and run for at least 5 minutes. Attach a rotameter, read the ball, adjust so that the ball reads 65, and seal the adjusting mech-

anism so that it cannot be changed easily. Shut off motor, remove the filter, and attach the orifice calibration unit in its place. Operate the high-volume sampler at a series of different, but constant, airflows (usually six). Record the reading of the differential manometer on the orifice calibration unit, and record the readings of the rotameter at each flow. Measure atmospheric pressure and temperature. Convert the differential manometer reading to m^3/min , Q , then plot rotameter reading versus Q .

8.1.3 Correction for Differences in Pressure or Temperature. See Addendum B.

9 Calculations

9.1 Calibration of Orifice

9.1.1 True Air Volume. Calculate the air volume measured by the positive displacement primary standard.

$$V_s = \frac{(P_s - P_a)}{P_a} (V_s)$$

V_s = True air volume at atmospheric pressure, m^3

P_s = Barometric pressure, mm. Hg.

P_a = Pressure drop at inlet of primary standard, mm. Hg.

V_u = Volume measured by primary standard, m^3

9.1.2 Conversion Factors.

Inches Hg. $\times 25.4$ = mm. Hg.

Inches water $\times 73.48 \times 10^{-3}$ = inches Hg.

Cubic feet air $\times 0.0283$ = cubic meters air.

9.1.3 True Airflow Rate.

$$Q = \frac{V_s}{T}$$

Q = Flow rate, m^3/min .

T = Time of flow, min.

9.2 Sample Volume

9.2.1 Volume Conversion. Convert the initial and final rotameter readings to true airflow rate, Q , using calibration curve of 8.1.2.

9.2.2 Calculate volume of air sampled

$$V = \frac{Q_i Q_f}{2} \times T$$

V = Air volume sampled, m^3

Q_i = Initial airflow rate, m^3/min .

Q_f = Final airflow rate, m^3/min .

T = Sampling time, min.

9.3 Calculate mass concentration of suspended particulates

$$SP = \frac{(W_i - W_f) \times 10^6}{V}$$

SP = Mass concentration of suspended particulates, $\mu g/m^3$

W_i = Initial weight of filter, g.

W_f = Final weight of filter, g.

V = Air volume sampled, m^3

10^6 = Conversion of g. to μg .

10 References

- (1) Robson, C. D., and Foster, K. E., "Evaluation of Air Particulate Sampling Equipment", *Am. Ind. Hyg. Assoc. J.* 24, 404 (1963).
- (2) Tierney, G. P., and Conner, W. D., "Hygroscopic Effects on Weight Determinations of Particulates Collected on Glass-Fiber Filters", *Am. Ind. Hyg. Assoc. J.* 28, 363 (1967).
- (3) Unpublished data based on a collaborative test involving 13 participants, conducted under the direction of the Methods Standardization Services Section of the National Air Pollution Control Administration, October, 1970.
- (4) Harrison, W. K., Nader, J. S., and Fugman, F. S., "Constant Flow Regulators for High-Volume Air Sampler", *Am. Ind. Hyg. Assoc. J.* 31, 116-120 (1960).

- (5) Pate, J. B. and Tabor, E. C., "Analytical Aspects of the Use of Glass-Fiber Filters for the Collection and Analysis of Atmospheric Particulate Matter", *Am. Ind. Hyg. Assoc. J.* 23, 144-150 (1962).

APPENDIX

A. Alternative Equipment.

A modification of the high-volume sampler incorporating a method for recording the actual airflow over the entire sampling period has been described, and is acceptable for measuring the concentration of suspended particulates (Henderson, J. S., Eighth Conference on Methods in Air Pollution and Industrial Hygiene Studies, 1967, Oakland, Calif.). This modification consists of an exhaust orifice meter assembly connected through a transducer to a system for continuously recording airflow on a circular chart. The volume of air sampled is calculated by the following equation:

$$V = Q \times T$$

Q = Average sampling rate, m.³/min.
T = Sampling time, minutes.

The average sampling rate, Q, is determined from the recorder chart by estimation if the flow rate does not vary more than 0.11 m.³/min. (4 ft.³/min.) during the sampling period. If the flow rate does vary more than 0.11 m.³ (4 ft.³/min.) during the sampling period, read the flow rate from the chart at 2-hour intervals and take the average.

B. Pressure and Temperature Corrections.

If the pressure or temperature during high-volume sampler calibration is substantially different from the pressure or temperature during orifice calibration, a correction of the flow rate, Q, may be required. If the pressures differ by no more than 15 percent and the temperatures differ by no more than 100 percent (°C), the error in the uncorrected flow rate will be no more than 15 percent. If necessary, obtain the corrected flow rate as directed below. This correction applies only to orifice meters having a constant orifice coefficient. The coefficient for the calibrating orifice described in 5.1.4 has been shown experimentally to be constant over the normal operating range of the high-volume sampler (0.6 to 2.2 m.³/min.; 20 to 75 ft.³/min.). Calculate corrected flow rate:

$$Q_c = Q_u \left[\frac{T_u P_c}{T_c P_u} \right]^{1/2}$$

Q_c = Corrected flow rate, m.³/min.

Q_u = Flow rate during high-volume sampler calibration (Section 5.1.2), m.³/min.

T_u = Absolute temperature during orifice unit calibration (Section 5.1.1), °K or °R.

P_u = Barometric pressure during orifice unit calibration (Section 5.1.1), mm. Hg.

T_c = Absolute temperature during high-volume sampler calibration (Section 5.1.2), °K or °R.

P_c = Barometric pressure during high-volume sampler calibration (Section 5.1.2), mm. Hg.

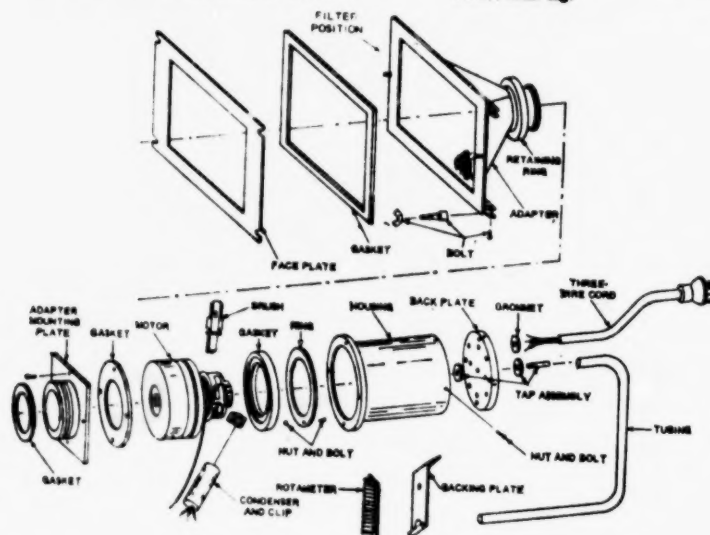


Figure B1. Exploded view of typical high-volume air sampler parts.

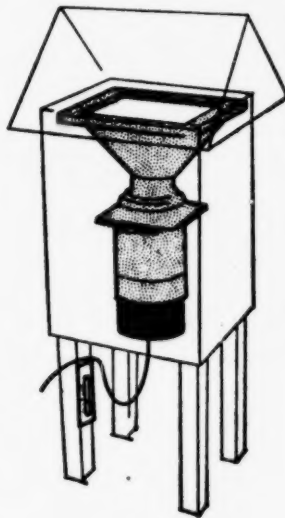


Figure B2. Assembled sampler and shelter.

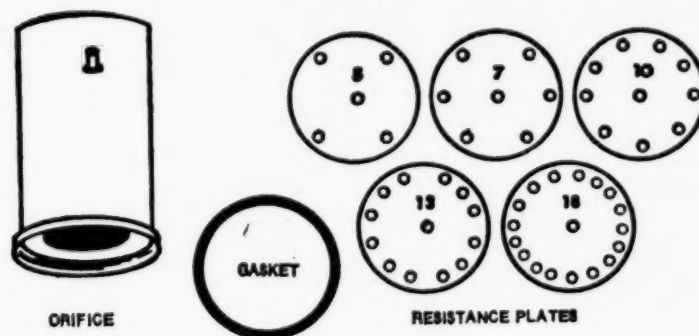


Figure B3. Orifice calibration unit.